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Performance Properties of Half-bleached Weft Knitted Fabrics Made of 100% Cotton Ring Yarns with Different Parameters

Učinkovitost lastnosti polbeljenih votkovnih pletiv, izdelanih iz 100-odstotnih bombažnih prstanskih prej z različnimi parametri

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Abstract

Knitted fabrics are distinguished by their outstanding comfort for clothing and for their rapid mass production. Though cotton knitted fabrics can provide better comfort, their physical appearance and service life are affected by many factors, and they have a propensity for pilling, abrasion and snagging. The main goal of this research work was to investigate the effect of yarn parameters on the abrasion, pilling and snagging resistance of half-bleached knitted fabrics. Six knitted fabrics were manufactured from 100% cotton carded ring yarn with a linear density of 21, 25, and 30 tex, with two yarn twist levels for each linear density. Except for yarn linear density and twist, the remaining yarn and machine parameters were constant, including fabric manufacturing. The knitted fabrics were treated using a half-bleach treatment before property evaluation. The results showed that knitted fabric made from a finer count of 21 tex with a higher yarn twist of 920 m^{-1} had the highest mass loss ratio of 2.12–10.76%, and the lowest abrasion resistance of 89–97.88% between 5,000 to 20000 abrasion cycles. The highest abrasion resistance of 96.4–98.9% (mass loss ratio of 1–3.5%) was recorded for a single jersey knitted fabric made from coarser yarn (30 tex) with the lower twist of 826 m^{-1}. The abrasion resistance of knitted fabrics was significantly affected by the thickness of the fabric, while regression analysis proved that fabric thickness and mass loss ratio had very good correlation, with an adjusted R^2 value of 93.8%. The snagging resistance of knitted fabrics increased as yarn twist and fineness increased. Pilling propensity increased as yarn linear density increased and twist decreased. Linear regression results revealed that yarn linear density and twist were highly correlated to abrasion resistance (mass loss method) at an adjusted R^2 value of 98.6% or 0.986 after 20,000 rubs.
Keywords: yarn parameters, knitted fabrics, half-bleached, abrasion, pilling, snagging resistance

Izvleček

Izjemna udobnost pletenih oblačil in hitra masovna proizvodnja pletiv sta znani. Čeprav bombažna pletiva zagotavljajo udobje, pa sta njihov estetski videz in doba trajanja odvisna od številnih dejavnikov, ki vodijo v nastanek pilinga, drgnjenje in izvlečenje zank. Glavni cilj te raziskave je bil proučiti vplive parametrov preje na odpornost proti drgnjenju,

pilingu in izvlečenju zank polbeljenih pletiv. Izdelanih je bilo šest pletiv iz 100-odstotnih bombažnih mikanih prstanskih prej s finočo 21 tex, 25 tex in 30 tex z dvema različnima stopnjama vitja. Preostali parametri izdelave prej in pletiv so bili enaki. Pletiva so bila pred oceno lastnosti polbeljena. Rezultati so pokazali, da je pletivo, izdelano iz finejše preje 21 tex z višjim vitjem 920 m^{-1}, pri 5000 do 20.000 ciklih drgnjenja izgubilo največ mase, in sicer 2,12–10,76 %, ter imelo najnižjo, 89–97,88-odstotno odpornost proti obrabi. Najvišja, 96,4–98,9-odstotna odpornost proti drgnjenju (masa zmanjšana za 1–3,5 %), je bila zabeležena pri enojnem jerseju, izdelanem iz preje 30 tex z vitjem 826 m^{-1}. Na odpornost proti drgnjenju je močno vplivala debelina pletiv. Regresijska analiza je pokazala, da sta debelina in zmanjšanje mase med seboj močno soodvisna, kar je potrdil 93,8-odstotni korelacijski koeficient, R^2. Odpornost pletiva proti izvlečenju zank se je povečala z vitjem in finočo preje. Nagnjenost k pilingu se je povečala, ko se je finoča preje zvišala in se je vitje znižalo. Rezultati linearne regresije po 20.000 drgnjenjih (metoda zmanjšanja mase) so pokazali korelacijo finoče in vitja preje z odpornostjo proti drgnjenju, saj je korelacijski koeficient, R^2, znašal 98,6 odstotka ali 0,986.
Ključne besede: parametri preje, pletivo, polbeljeno pletivo, drgnjenje, piling, odpornost proti izvlečenju zank

1 Introduction

Abrasion resistance and pilling performance are two of the most important mechanical characteristics of fabrics [1] and a factor in virtually every textile application. They are also a major purchasing requirement from the consumer's viewpoint. The abrasion resistance of textile materials is affected by many factors in a very complex and poorly understood manner [2]. Market studies have shown that evaluations of consumer quality requirements are related to abrasion resistance [3]. Abrasion is the gradual removal of fibres from yarns, and is influenced by fibre cohesion in yarns [4]. Kalaoglu et al. and McCord [3, 5] stated that the abrasion resistance of textile materials is affected by many factors, such as fibre content, fibre fineness, yarn linear density, yarn type, weave, fabric thickness, finishes, etc. Abrasion first modifies the fabric surface and then affects the internal structure. Similarly, the pilling of knitted fabrics is a persistent and serious problem for the clothing industry [6]. Many parts of clothing, such as collar, cuffs, and pockets are subjected to wear in use, which limits their serviceability. Pilling not only reduces appearance and comfort properties, but also affects the service life of textile products [7]. Uyanik and Topalbekiroglu studied the effects of knit structures on the pilling resistance of knitted fabrics made from the same cotton yarn. The results revealed that single jersey has a lower pilling resistance than fabrics with tuck stitches, while knit structures with larger pores show higher a resistance to pilling. Some authors studied the relationship between fibre, yarn and wool single jersey and rib knitted fabrics on pilling property. The prediction of the pilling tendency of those wool knits was developed by artificial neural network modelling (ANN) [8, 9].

In addition, dyeing and finishing processes reduce the pilling resistance of knitted fabrics [10]. Other authors have researched the effect of wet processing on cellulosic knitted fabrics, and the model suggests that the ends of fibres come out from yarns by mechanical abrasion due to low fibre-fibre friction [11]. Candan studied the pilling and abrasion properties of different knitted fabrics made from ring and open-end spun cotton yarns, and 50/50 cotton/polyester yarns. The results showed that, unlike plain jersey fabrics, Lacoste fabrics perform very well and that fabrics knitted from open-end spun yarns generally have a lower propensity to pilling [12]. Akaydin and Can stated that the abrasion resistance and pilling performance of interlock fabrics were higher than jersey fabrics, those of dyed fabrics higher than raw fabrics, and those fabrics produced from compact yarns were higher than fabrics produced from ring yarns [13]. Another researcher investigated the effects of fibre type, and single and ply yarns on the abrasion and pilling resistance of socks. The results indicated that the abrasion resistance of socks increased with use of coarser yarn or thicker yarn, the addition of polyester, and the addition of polyamide or elastic yarns to the structure [14]. Knitted fabrics with interlock, rib and single jersey structures made from compact and conventional ring yarns and their physical properties were investigated, and compared with each other before and after printing processes. It was found that no statistical differences were observed with regard to weight, abrasion resistance, colour efficiency and rubbing fastness [1].

A previous study reported that the fibrous composition and thickness of materials (up to 6%), as well as washing and softening (from 33% to 67%) change the pilling resistance of knitted fabrics [15]. Daiva studied the pilling resistance of single jersey, rib and in-

terlock knitted fabrics made from PES yarns, cotton yarns and cotton yarns combined with PU yarns. The results found that 2×2 rib knitted fabric has a better pilling resistance than interlock, 1×1 rib and plain knitted fabric because of the reduced operating surface area. Fabrics knitted from PES (polyester) yarns or those PES yards blended with cotton yarns have a worse visual appearance than fabrics knitted from pure cotton yarns because PES fibres are resistant to malformed pills due to their exceptional strength [6]. A 100% cotton single jersey knitted fabric made from a combed ring yarn with a linear density of 19.7 tex, and a different stitch position and length, was studied, and the results revealed that stitch density (wales/cm and course/cm) and mass per unit area, bursting, pilling and abrasion resistance decrease as stitch length increases [16, 17]. Single jersey knitted fabrics with various numbers and position of tuck stitches were made from glass yarn using a flat knitting machine. The findings proved that fabric thickness increased and air permeability decreased as tuck stitches increased [18]. The effect of rubbing fastness on single jersey knitted fabrics made of combed ring-spun and compact cotton yarns was researched. The reports showed that knitted fabrics made from compact and comb yarn were similar in terms of rubbing property in a dry state. However, single jersey knitted fabrics made from ring yarn have a greater rubbing resistance in a wet state due to the high porosity of the ring yarn fabrics, which allows water molecules to penetrate [19, 20].

Numerous researchers have stated that the end-use properties of clothing, such as pilling effect and abrasion resistance, are influenced by material type, fibre fineness, yarn linear density, yarn strength, yarn hairiness, fabric structure and surface density [2, 21–24]. A comparison of pilling and abrasion properties of knitted fabrics was performed for different spinning yarns, such as ring, compact and rotor yarns [25–30]. The effects of washing and drying [1] as well as finishing and dying [31, 32], were also investigated. Knitted fabrics from compact yarns have a higher pilling and abrasion resistance than ring and rotor yarns, while the abrasion resistance of knitted fabrics from ring-spun yarns was slightly better than open-end spun yarns [33].

As shown in a review of literature, many researchers have studied the effects of yarn structure, linear density, twist and knitted structure on the abrasion, pilling, tensile and tear strength properties of fabrics. Most of the studies were done at the greige fabric level and based on a comparison based on different spinning methods. However, the effects of yarn properties on fabric snagging resistance have not been thoroughly addressed. The aim of this study was to investigate the influence of ring yarn parameters (by varying yarn linear density and twist) on the pilling, abrasion and snagging resistance of half-bleached knitted fabrics.

2 Materials and methods

2.1 Materials

Six single jersey knitted fabrics were produced from 100% cotton carded ring yarn counts of 21 tex with two twist levels of 920 m^{-1} and 905 m^{-1}, 25 tex with twist levels of 890 m^{-1} and 860 m^{-1}, and 30 tex with twist levels of 847 m^{-1} and 826 m^{-1}.

2.2 Methods

Each of the three yarns with two different twist levels were produced using a ring spinning system (RIETER-G35) manufactured by Bahir Dar Textile Share Company. All yarns were spun from the same fibre mix with a micronaire value of 4.23, a maturity of 0.85, an upper half mean length (UHML) of 29.86 mm, a uniformity index (UI) of 84.5%, a strength of 30.4 cN/tex, an elongation of 7.4%, a short fibre content of 6.7% and a trash grade of three. Yarn linear density and yarn twist were measured according to the ES ISO 2060 and ASTM D1422 test methods, respectively. Six knitted fabrics were manufactured using those developed yarns with incremental twist levels. Except for yarn linear density with different twist levels, the remaining parameters were constant and the knitted fabrics were produced on the same SHIMA SEIKI® (model – SES 122 FF, gauge 7) flat knitting machine at the Technical University of Liberec in the Czech Republic. The machine settings, such as machine speed, loop length, and horizontal and vertical density per centimetre were kept constant for all fabrics. The yarn and fabric characteristics are presented in Table 1.

Chemical treatments

Hydrogen peroxide (H_2O_2) based half-bleach combined treatment was carried out for knitted fabrics using a winch machine. The fabric and water solution were prepared at a material to liquor ratio (MLR) of 1:5, and hydrogen peroxide (H_2O_2), sodium silicate (Na_2SiO_3), sodium hydroxide (NaOH) and a wetting

agent of 4%, 2%, 3%, and 0.5% of fabric weight, respectively. Knitted fabrics were treated at a temperature of 95 °C for 90 minutes at a machine working speed of 40 m/minutes.

Abrasion resistance

The abrasion resistance of the knitted fabrics was measured using two methods according to ES ISO 12947-1 (appearance change method) at 5,000 cycles and ES ISO 12947-3 (mass loss method) using a Martindale Abrasion and Pilling device (Mesdan-Lab, Model 2568). The mass loss ratios of knitted fabrics were recorded after 5,000, 7,500, 10,000, 15,000 and 20,000 cycles of the Martindale Abrasion and Pilling device. Pressure loading of 9 kPa was used during testing for both methods (1 and 3).

Pilling resistance

The pilling properties of knitted fabrics were measured using two different methods according to ES ISO 12945-2 after 5,000 cycles using a Martindale Abrasion and Pilling device. The second evaluation was based on ES ISO 12945-1 at 18,000 cycles according to the ICI Pilling-Box method using a Mesdan-Lab device, model no. 1006). Finally, the pilling grade was rated using the EMPA (SN 19825) photographic standard.

Snagging resistance

A snagging test was evaluated according to ASTM D3939 after 600 revolutions using an SDL ICI Mace snagging tester (model no. P22668). The test specimens were graded using the relevant photographic standard.

Statistical analysis

To determine the analysis of variance and significant test, SPSS version 25 for Windows statistical software was used. Regression analysis was performed using Origin Lab software (version 9.6.5) to determine the correlation of factors (yarn parameters) and response (fabric properties).

3 Results and discussion

Table 1 presents the properties and characteristics of the yarns used and the developed knitted fabrics. Except for yarn count and twist levels, all knitted fabrics were produced using the same yarn property, knit type and thread density.

Structural properties of knitted fabrics

The structural parameters of the knitted fabrics, such as stitch density, loop length, cover factor, thickness and fabric weight were tested and the results are presented in Table 1.

The fabric cover factor K is defined as the proportion of the fabric area covered by actual yarn and expressed using equation 1.

$$K = \frac{\sqrt{\mathrm{Tt}}}{\ell} \tag{1}$$

where T_t represents linear density (tex) and ℓ represents loop length.

Effects of yarn parameters on abrasion resistance (appearance change)

As seen in Figure 1, the abrasion resistance (appearance change) of knitted fabrics declined from fabric K1 to K6. This is probably because individual fibres and neps were not firmly held in the yarn cross-section as turns per meter were decreased. Knitted fabrics made from lower twist yarns had a significant abrasion effect (appearance change) since the

Table 1: Yarn and knitted fabric characteristics

Fabric code	Yarn linear density (tex)	Twist (m^{-1})	Knit type	Loop length (mm)	Cover factor (K)	Stitch density (stitch/cm) (horizontal/ vertical)	Thickness (cm)	Mass per unit area (g/m^2)
K1	21	920	Plain	4.10	0.96	9/12	0.071	184
K2	21	905	Plain	4.10	0.98	9/12	0.075	189
K3	25	890	Plain	4.12	1.21	9/12	0.082	196
K4	25	860	Plain	4.11	1.26	9/12	0.086	203
K5	30	847	Plain	4.10	1.37	9/12	0.091	211
K6	30	826	Plain	4.08	1.44	9/12	0.096	224

presence of hairiness and neps were high in lower twist yarns. As is evident from Figure 1, fabrics K4, K5 and K6 from coarser yarn counts of 25 tex and 30 tex with lower twists of 860 m^{-1}, 847 $m^{-1}$and 826 m^{-1} had a slightly poor abrasion resistance grade in surface appearance. As stated above, this is because fibres in the yarn cross-section are not held firmly and are thus easily pulled by the abradant. As seen in Table 2, the statistical analysis also confirmed that abrasion resistance in the appearance change method resulted in a significant change at an F-value of 13.000 and P-value of 0.033. If the P-value is greater than 0.05, this means the samples have similar properties, while the opposite is true if the P-value is less than α value of 0.05.

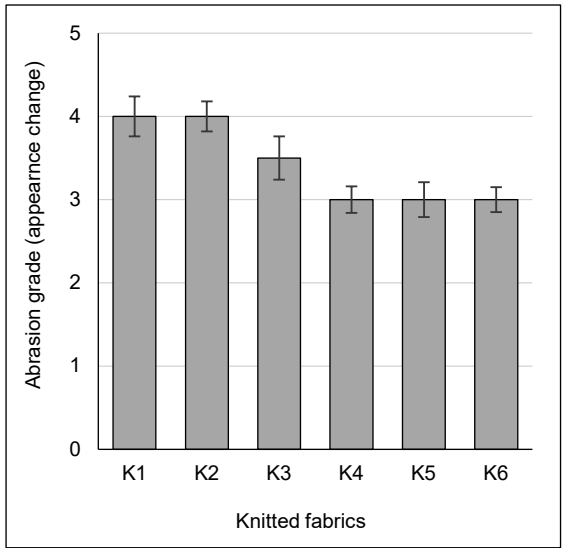

Figure 1: Abrasion resistance of knitted fabrics using the appearance change method

Twist is an important parameter affecting abrasion. At low twist levels, it was observed that fibres can be easily pulled from the yarn cross-section and that the resistance grade was reduced at 5,000 rubs. At high twist levels, however, the fibres are held more tightly, but the yarn is stiffer, so it is unable to distort under pressure when it is abraded. The findings of this study are in line with Saville's report [4]. Multiple regression analysis showed that the studied yarn parameters have a positive correlation with the abrasion resistance (appearance change) of knitted fabrics at an adjusted R^2 of 0.801. The adjusted R^2 value is an indication of the correlation of yarn properties (factors) and fabric characteristics (responses). When the adjusted R^2 value increases or decreases to 1 or –1, this indicates a strong correlation between them [34].

Effects of yarn parameters on abrasion resistance (mass loss)

As is evident from Figure 2, the knitted fabric weight mass loss ratio was increased as abrasion cycles increased in all fabrics. Nevertheless, the abrasion resistance increased as yarn count (tex) increased and yarn twist decreased. On the other hand, finer counts and higher twist yarns resulted in fabrics with thinner thickness, which had a high tendency to be abraded quickly. This, in turn, this led to higher mass loss ratio. As seen in Figure 2, knitted fabrics made from 30 tex with a yarn twist of 847 m^{-1} have a higher abrasion resistance of 96.4–98.9% (mass loss ratio of 1–3.5%) between 5,000 to 20,000 abrasion cycles than other developed fabrics. Knitted fabrics made of 25 tex with a twist per meter (TPM) of 890 m^{-1} and 860 m^{-1} demonstrated moderate abrasion

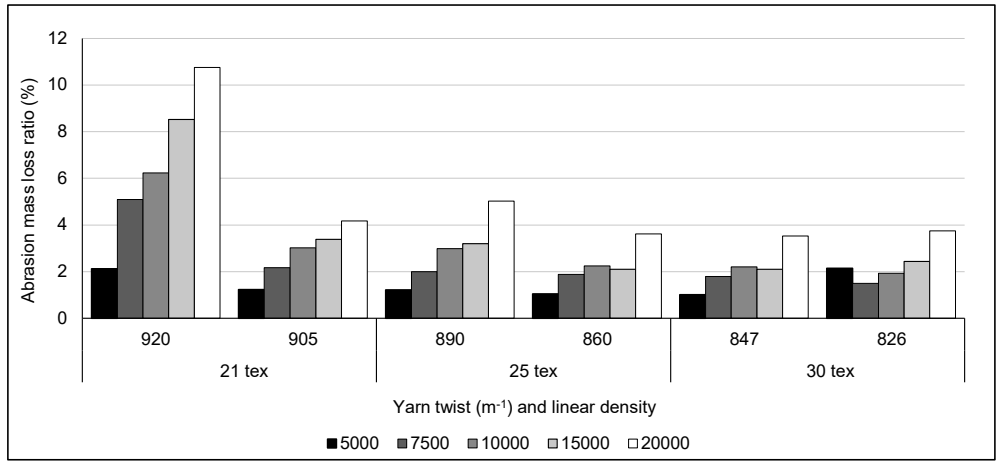

Figure 2: Abrasion results of knitted fabrics using the mass loss method (at 5,000, 7,500, 10,000, 15,000 and 20,000 rubs)

resistance, while knitted fabrics made from a finer yarn count of 21 tex with the highest yarn twist of 920 TPM demonstrated the highest mass loss ratio of 2.12–10.76% or an abrasion resistance of 89–97.88%. For this reason, fabrics from coarser yarn are thicker and bulky, and will require a great deal of time and a higher number of abrasion cycles to lose their original mass. These findings inveterate an earlier report by Kalaoglu and Onder [5].

As illustrated in Table 2, the statistical analysis proves that the abrasion resistance (mass loss method) of knitted fabric resulted in a insignificant change at a P-value of 0.660 after 5,000 rubs, and a significant change at P-values of 0.038, 0.000, 0.010 and 0.008 after 7,500, 10,000, 15,000 and 20,000 rubs, respectively. The results showed that the correlation of yarn parameters and abrasion resistance (mass loss method) was low at an adjusted R^2 value of 0.242 after 5,000 rubs. On the other hand, yarn twist and count were highly correlated with abrasion resistance at an adjusted R^2 value of 98.6% or 0.986 after 20,000 rubs. As observed from Table 1 and Figure 2, fabric thickness affects the abrasion resistance of knitted fabrics because thin fabrics withstand damage during friction for an extended period, and vice versa for thicker fabrics. The regression analysis proves that fabric thickness and abrasion mass loss are directly proportionate and correlated with an adjusted R^2 value of 84.3%.

Effects of yarn parameters on pilling resistance

Table 3 illustrates the pilling resistance of knitted fabrics using the Martindale and ICI Pilling Box methods. The results obtained from the ICI Pilling Box method demonstrated a higher pilling resistance than the pilling grade using the Martindale method for all knitted fabrics. This is because the ICI Pilling Box method was performed at a low mechanical force, while test specimens were dropped randomly on a wooden board during rotations. However, mechanical force is higher in the Martindale tester than in the ICI Pilling Box method because consistent friction is formed by the Lissajous pattern form on the fabric (ISO 12947-1: 1998). In both test methods, knitted fabrics made from finer ring yarns had a higher pilling resistance than fabrics from coarser yarns. The reason for this is that the coarser yarns had less twist and fibres are easily pulled from the yarn cross-section. On the contrary, knitted fabrics from finer yarns had a compact structure, which means fibres would be hidden by twist and are hard to raise by the piler. The researchers Omeroglu and Ulku also reported a similar concept [2].

Table 2: Analysis of variance of knitted properties

Fabric properties		Sum of squares	Df	Mean square	F	Sig.
Pilling (Martindale method)	Between groups	0.333	5	0.167	1.333	0.385
	Within groups	0.375	3	0.125		
Pilling (ICI Box method)	Between groups	0.583	5	0.292	13.500	0.004
	Within groups	0.250	3	0.083		
Abrasion resistance (appearance change)	Between groups	1.083	5	0.542	13.000	0.033
	Within groups	0.125	3	0.042		
Abrasion mass loss (at 5,000 rubs)	Between groups	0.336	5	0.168	0.478	0.660
	Within groups	1.054	3	0.351		
Abrasion mass loss (at 7,500 rubs)	Between groups	4.606	5	2.303	11.589	0.038
	Within groups	4.348	3	1.449		
Abrasion mass loss (at 10,000 rubs)	Between groups	7.293	5	3.646	12.006	0.000
	Within groups	5.454	3	1.818		
Abrasion mass loss (at 15,000 rubs)	Between groups	16.427	5	8.214	8.773	0.010
	Within groups	13.896	3	4.632		
Abrasion mass los (at 20,000 rubs)	Between groups	16.695	5	8.347	11.102	0.008
	Within groups	22.724	3	7.575		
Snagging resistance	Between groups	3.083	5	1.542	18.500	0.021
	Within groups	0.250	3	0.083		

Table 3: Pilling resistance of knitted fabrics

Yarn linear density (tex)	Fabric code	Evaluation methods	
		Martindale method	ICI Pilling Box
21	K1	3/4	4–5
	K2	3	4
25	K3	3	4
	K4	3/4	3–4
30	K5	3	3–4
	K6	2/3	3–4

As is evident from Table 2, the pilling resistance of knitted fabrics in the ICI Pilling Box method resulted in a significant change at an F-value of 13.500 and P-value of 0.004. On the other hand, knitted fabrics did not show a significant difference after the pilling property test using a Martindale Abrasion and Pilling device with an F-value of 1.333 and P-value of 0.385. In previous studies, some researchers reported a similar concept, i.e. pilling tendency increased as mass per unit area of polyester-cotton blended fabrics increased. [35, 36]. As mentioned above, multiple regression results proved that the pilling resistance of knitted fabrics (ICI Pilling Box method) had a negative correlation with studied yarn parameters with an adjusted R^2 value of -0.760.

Effect of yarn parameters on snagging resistance

Snagging resistance was evaluated using ICI photographic snagging standards, which consists of five incremental photo replicas, grades 5 to 1. Grade 5 indicates no snagging, grade 4 indicates slight snagging grade 3 indicates moderate snagging, grade 2 indicates sever snagging and grade 1 indicates very severe snagging. As shown in Figure 3, the snagging resistance of knitted fabrics decreased as yarn linear density (tex) increased and twist decreased. Knitted fabrics K1 and K2 made from 21 and 25 tex had a higher snagging resistance (grade 4–5), followed by fabric from 30 tex. The snagging grade also declined as yarn twist decreased. This is because knitted fabrics from higher twist yarns are very strong and fibres are held firmly in the yarn cross-section, meaning they are not easily snagged by sharp and rough objects. As stated in a previous study, yarn strength generally increases as yarn twist increases (19). However, fabrics made from coarser yarn with a lower twist demonstrated a poor snagging grade because yarns with less yarn twists have low turns per meter in the yarn cross-section, and are more easily snagged by sharp materials. These findings confirm an earlier study by Paek [7].

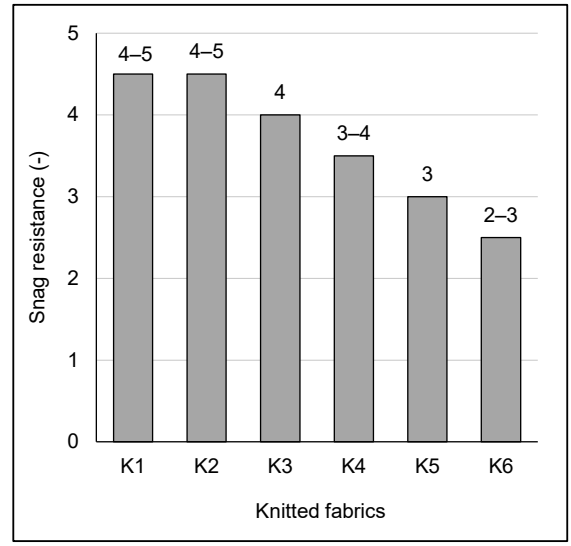

Figure 3: Snagging resistance

As shown in Table 2, the snagging resistance of knitted fabrics was affected by yarn parameters at an F-value of 18.500 and P-value of 0.021. Multiple linear regression proved that yarn parameters had very good correlation with the snagging resistance of fabrics at an adjusted R2 value of 0.949 (94.9%). In addition, yarn count and fabric thickness were highly correlated with the snagging resistance of knitted fabrics at an adjusted R2 value of 0.898 (89.8%) and 0.955 (95.5%), respectively. Textile materials, such as knitted fabrics and military cloths, are subjected to snagging. Rough objects, fingernails or toenails are some initiators of the snagging effect for knitted fabrics. Therefore, knitted fabric manufacturers should consider yarn count and twist for the desired snagging resistance of fabrics.

4 Conclusion

Six 100% cotton single jersey knitted fabrics were produced from 21, 25 and 30 tex ring-spun yarns with different twist levels. The knitted fabrics underwent half-bleach treatment and drying. The abrasion, pilling and snagging properties of knitted fabrics were evaluated using a Martindale Abrasion and Pilling tester, the ICI Pilling Box method and a Mace snagging tester. The results obtained showed that knitted fabric made from a finer count of 21 tex with highest yarn twist of 920 TPM demonstrated the highest mass loss ratio of 2.12–10.76% (poor abrasion resistance 89–97.88%) between 5,000 to 20,000 abrasion cycles. On the contrary, single jersey knitted fabrics made from coarser yarn (30 tex) with the lowest twist (826 TPM) demonstrated a higher abrasion resistance of 96.4–98.9% (mass loss 1–3.5%). The pilling propensity increased as yarn count (tex) increased and twist decreased. Linear regression results revealed that yarn count and twist were highly correlated with abrasion resistance (mass loss method) at an adjusted R^2 value of 98.6% or 0.986 after 20,000 rubs. The snagging resistance of knitted fabrics increased as yarn twist and yarn fineness increased. Generally, abrasion resistance was highly affected by the thickness of the fabric, while regression analysis proved that fabric thickness and mass loss ratio had very high correlation with an adjusted R2 value of 93.8%.

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Optimisation in the Logistics and Management of Supply Chains in Production by Textile Enterprises

Optimizacija v logistiki in upravljanju dobavnih verig proizvodnje v tekstilnih podjetjih

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Abstract

This article is devoted to questions regarding the analysis of the implementation of logistics and supply chain management conditions in textile production. Based on delivery optimisation, the authors offer a model of multimodal transportation of textile products produced in Uzbekistan. The importance of optimising the supply chain of the logistics business processes in order to decrease costs is demonstrated in this article. A mathematical model of optimisation for placement textile enterprises to stimulate the reduction of supply chain costs is recommended. However, this research would be beneficial for the textile and fashion industries. The approach might be further extended to other similar industries.
Keywords: logistics, transportation management, multimodal transportation, optimisation, supply chain management

Izvleček

V članku so obravnavana vprašanja, povezana z analizo pogojev izvedbe logistike in upravljanja dobavne verige v tekstilni proizvodnji. Na podlagi optimizacije dostave avtorji predlagajo kombinirane prevoze tekstilnih izdelkov, izdelanih v Uzbekistanu. V članku je dokazan pomen optimizacije dobavne verige logističnih poslovnih procesov za zmanjšanje stroškov. Priporočen matematični model optimizacije plasiranja tekstilnih podjetij spodbuja zmanjšanje stroškov dobavne verige. Raziskava se nanaša na tekstilno in modno industrijo, vendar je pristop mogoče razširiti na druge podobne industrije.
Ključne besede: logistika, upravljanje prevoza, kombinirani prevozi, optimizacija, upravljanje dobavne verige

1 Introduction

The globalisation of the world economy, the development of information technologies, means and ways of delivering products, the outsourcing of business processes, and services have a considerable impact on the way administrative decisions are adopted in all components of the business processes of production,

marketing, commerce and logistics [1]. Adopting optimal administrative solutions in difficult economic situations has always been a constant in the practical activities of textile sectors throughout the world [2]. Moreover, their role has increased considerably recently as the dynamism of the external and internal environment has increased. The period required to make decisions has been reduced. The development of science and technologies has resulted in the emergence of many alternative options and interdependence. Different administrative decisions and their consequences have been amplified. The labour input required to adopt and implement challenging and multi-criteria decisions has increased significantly [3]. In these conditions, establishing rational and optimal solutions is the main focus of developing textile enterprises' logistics and organisations' business processes at the strategic and operational levels to improve supply chain management and logistics methods [4, 5]. The need for the high-quality growth of Uzbekistan's economy assumes that textile enterprise managers make better use of the entire range of methods and models of adopting optimal solutions in the supply, production and distribution of goods and services in the logistics and supply chain.

A diagnostic analysis of the administrative decisions made by the managers of textile enterprises and supply chain participants allowed us to establish that adopting logistic decisions used in practice is characterised by utility and subjectivity, and a lack of modern computer technologies (software products). The conducted research can be deemed the further development of the theory and methodical bases of supply chain management, and an opportunity for the broader application of mathematical models and methods for adopting optimal logistic solutions in the performance of management functions and the business processes of production, distribution, transportation and consumption of intermediate and readymade products [6–10].

Despite the extensive and practical application of logistics and supply chain management in the organisation of the transportation and production of goods, it is still not fully implemented in Uzbekistan. There is a lack of exhaustive scientific research successfully carried out in these areas. The importance of optimising logistics business processes to cut costs is demonstrated in this article using a mathematical model. Though different models have been proposed for other industries, the textile and fashion industry has not considered them. We have developed a method for optimising the business process of distribution and sales (supply) of a textile enterprise's finished products based on an economic and mathematical model for optimising the sales structure. Thus, this research practically presents a practical solution for both the textile and fashion industries.

2 Methods

2.1 Analysis of conditions in the textile industry in Uzbekistan

Uzbekistan is one of the largest global cotton-fibre suppliers, while it also pays a great deal of attention to the deep processing of raw cotton [11]. For instance, the current coefficient of processing is 40%. The adopted modernisation programme of the textile industry is expected to bring the processing volume to 70% by 2020 [5]. In the modern world, the textile industry possesses a high rating among the other exports. It has the broadest range of exported goods' nomenclature, from yarn to readymade goods (apparel and knitted products; see Figure 1).

The textile industry is an essential, versatile and innovatively attractive sector of the economy of Uzbekistan. Its role is a macroeconomic complex that can be assessed from the following data: the textile industry accounts for 2.7% of Uzbekistan's GDP, 26.2% of industrial output in terms of volume, and more than 34% of the production of non-food consumer goods. Four hundred textile companies equipped with modern conditions are included in the UZTEX Group. Of those, 130 are joint ventures created with the participation of foreign partners from the world's leading countries. The group records annual increases in production and exports of more than 18% and 10%, respectively. The annual combined output of group companies is around 480 thousand tonnes of yarn, 290 million square meters of cotton fabrics, 101 thousand tonnes of knitted cloth, 275 million pairs of stitched-knitted products, 53.1 million pairs of socks and hosiery, and 2.1 thousand tonnes of raw silk threads. Group companies also make products for medical use, nonwoven fabrics, batting products, uniforms and fashion apparel, and eiderdown products. Companies operate continuously using modern and efficient equipment. More than 1.6 million spinning spindles and 100 thousand cabinets are commissioned for operation, accounting for 89.3% of existing technological equipment. Products produced by the textile industry are exported to more

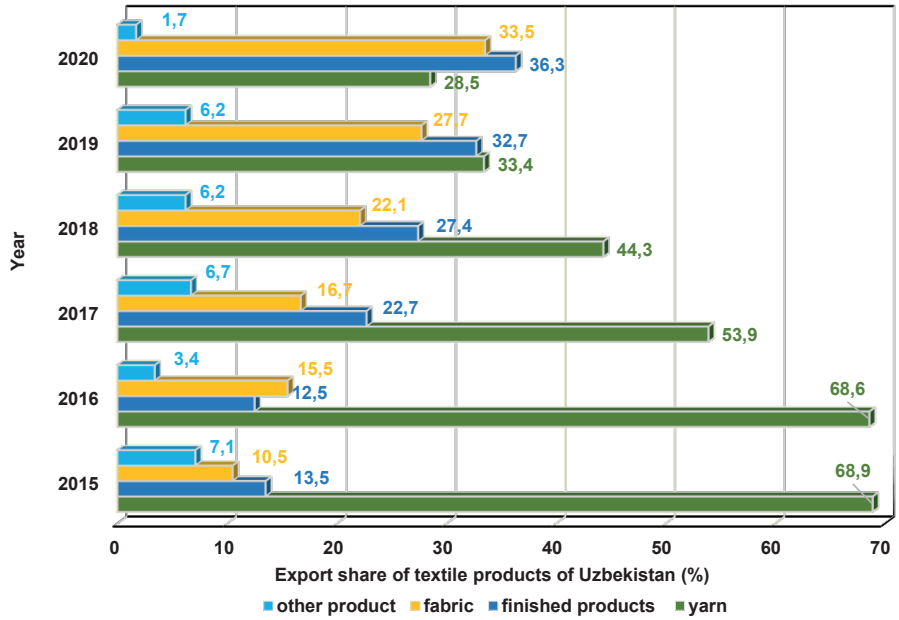

Figure 1: Structure of exports of textile products of Uzbekistan

than 50 countries, including European countries, China, the Commonwealth of Independent States, Latin America, the Republic of Korea, Singapore, Israel, Iran, the USA and others. In this regard, textile enterprises' supply chain management issues are crucial today.

The formation of textile supply chains has some sophisticated and distinctive influencing factors. They include the need for technological associativity based on a material stream that defines the contractors' choice, providing the delivery of and ability to render additional services. They also include the physical characteristics of a material stream that define a means of transportation and storage conditions, with the choice of the transport scheme and the warehousing method, respectively (refer, Figure 2).

One of the primary operating conditions of a textile enterprise's supply chain management is its interconnected system, which streams of goods and services and the labour force, and moves within the

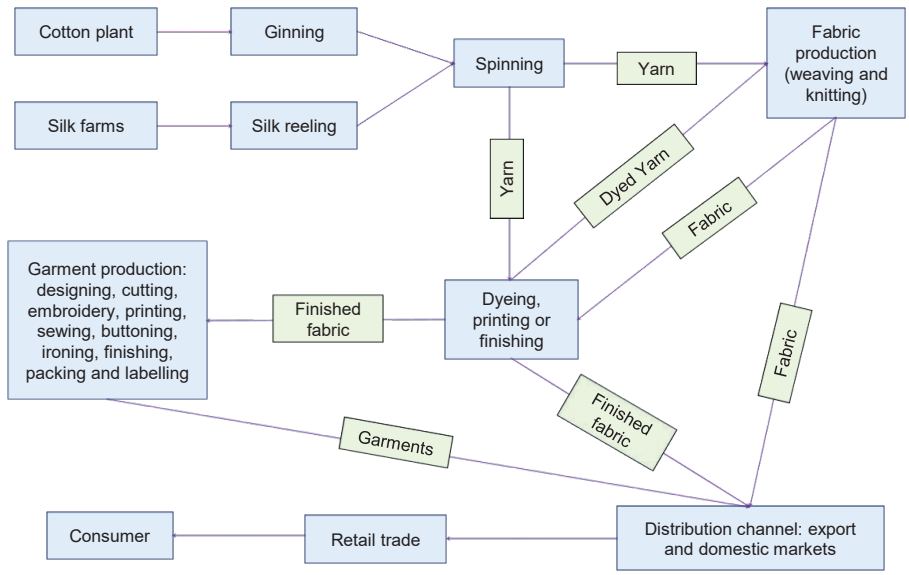

Figure 2: Formation of supply chains in a textile complex

market system under the influence of market stimulus [12–17].

2.2 Model formulation

An analysis of scientific literature over the last ten years regarding developments and the functioning of supply chains allowed us to formulate the following basic principles for carrying out the optimisation of supply chains and logistics business processes [18]:

I. The purposes of optimisation must be measurable and correspond to the optimality criteria of the actual logistic decision. This must be reflected in the statement of the corresponding task (the administrative decision). The following optimality criteria can be sued to create the general economic-mathematical model of optimisation of the sales of a manufacturer's finished goods: to maximise sales and profit, while minimising used resources and costs. Thus, various statements of problems of optimisation and the implementation of the associated economic-mathematical models can be developed. Thus, if the global purpose of business management lies in the hierarchy, then its purpose is about maximising overall profit, while the optimisation of supply is carried out for commodity groups of finished goods. To that end, the economic regulations of the sales dynamics of finished goods in natural units of measurement (O), proceeds from the sales of finished goods (R) and the dependence of price on sales of finished goods are used (C) in the implementation of these tasks. The graphs of these dependencies are shown in Figure 3.

$$C = a - b \times O \rightarrow O \times C = a \times O - b \times O^2 \quad (1)$$

$$O \times C = R \rightarrow R = a \times O - b \times O^2 \quad (2)$$

Consequently, a textile enterprise's profit as the difference between revenue and total costs (I) takes the form of a second-degree polynomial (P), which should be reflected in the profit maximisation problem statement.

Therefore, the profit of a textile enterprise, as a variety of procedures and general costs, has the aspect of a second-degree polynomial that must be reflected in a problem statement of maximising profit. The development of an economic-mathematical model for optimising the delivery of finished goods to commodity groups is based on the textile UZTEX Group. Optimality criteria result in the statement and solution of optimising the sales structure and delivery of finished goods to maximise a textile company's profit. Thus, the regression dependence of the profit of in one thousand US dollars from sales of t-shirts in one thousand pieces has the following aspect:

$$f(x) = 612.5x - 12.25x^2 \quad (3)$$

The regression dependence of the profit of in one thousand US dollars from sales of sportswear in one thousand pieces has the following aspect:

$$\varphi(x) = 82.2x - 1.1x^2 \quad (4)$$

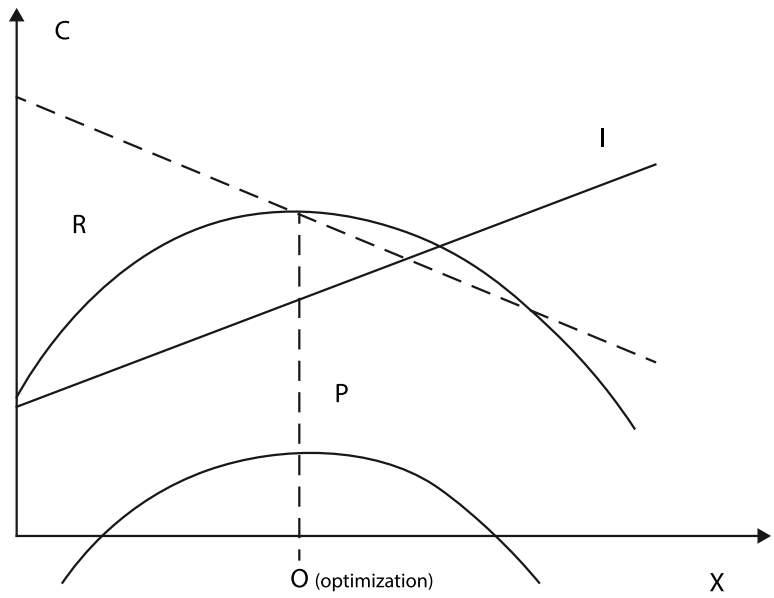

Figure 3: Dependence of the profit of a textile enterprise on product sales

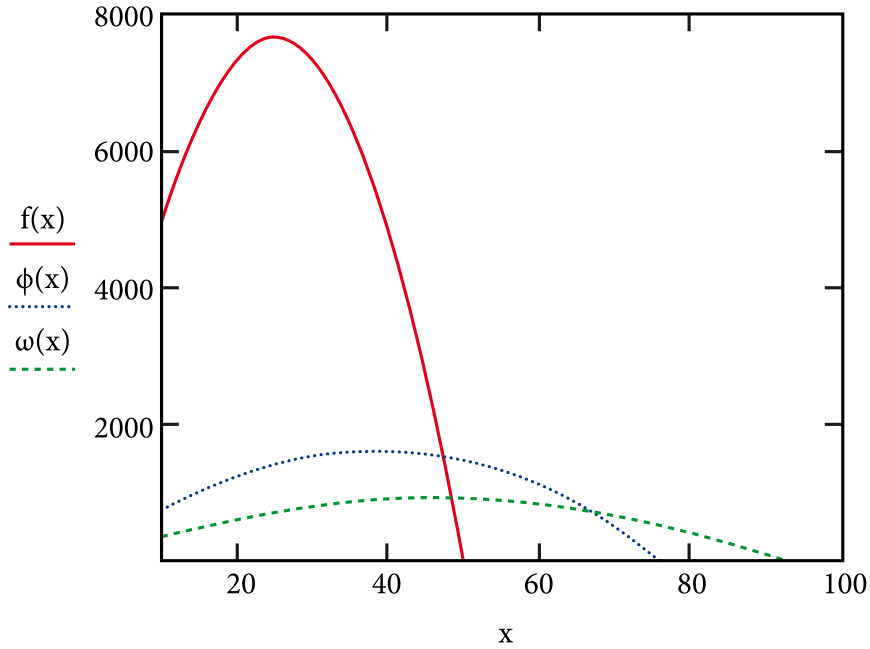

Figure 4: Regression dependences of profit from sales of finished products

The regression dependence of the profit of in one thousand US dollars from sales of hosiery in one thousand pieces has the following aspect:

$$\omega(x) = 38.9x - 0.42x^2 \tag{5}$$

The graphs of these regression dependences of profit from sales are shown in Figure 4. Graphical modelling allows us to conclude that there are maximum profit values at the optimal sales of the indicated types of finished products of a textile company. It is possible to optimize the structure of its sales and supply. The economic-mathematical modelling of the sales structure of finished goods of three main types consists of the formation additive function, which maximises the general profit from sales of t-shirts, sportswear and hosiery. The statement and solution of the optimising task are given below:

$$F = (x_1, x_2, x_3) = 612.5x_1 - 12.25x_1^2 + 84.2x_2 - 1.1x_2^2 + 38.9x_3 - 0.42x_3^2 \tag{6}$$

$$x_1 = 1;\ x_2 = 1;\ x_3 = 1 \tag{7}$$

Given $x_1 > 24\ x_2 > 32\ x_3 > 35$

$$Maximize(F, x_1, x_2, x_3) = \begin{bmatrix} 25 \\ 38 \\ 46 \end{bmatrix} \tag{8}$$

$$F(25, 38, 46) = 10170 \tag{9}$$

Thus, an optimal volume of the sale and supply of t-shirts to consumers is 25 thousand pieces, while that figure is 38 thousand pieces for sportswear and 46 thousand piecesfor hosiery. The UZTEX Group's overall maximum profit will be equal to $10.17 million.

II. Models of optimisation must be adequate, and accurately illustrate the logistics business processes and functions of supply chain management. Economic-mathematical optimisation models must be designed using concrete figures for logistics business processes and contain quantitatively measurable conditions for their implementation. They must be expressed in a system of restrictions of the model in terms of the size of used resources and reasonable assumptions on the scope of variation. In these terms, the research of operations applied in optimising logistics business processes should be supplemented with mathematic-statistical characteristics that take into account the probability of realisation under the established law of parameter distribution of these processes as random sizes.

III. The external conditions and parameters of the internal environment of supply chains and logistics business processes vary. While carrying out the optimisation, it is necessary to consider possible changes in the external conditions and parameters of logistic decisions. Similar changes are made periodically or

in the process of detection to the developed economic-mathematical models of optimisation. Previous practice with optimising models shows that they can be applied in an imitating form [19]. This assumes the automatic recalculation of optimisation results when there is a change of factorial signs and system parameters of the restrictions imposed on the used resources in supply chains.

IV. Data regarding the parameters of supply chains must be exact, timely and quick. This requirement is due to the use of that data in optimisation models, whose results significantly vary depending on the values of factorial signs and system of restrictions. Testing the results of the optimisation parameters of supply chains is obligatory. Such testing is carried out by verifying developed models and the results obtained via other economic-mathematical processing. The large number of records regarding supply chain parameters requires the preliminary analysis of those records, in a subsequence of integration and the use of new software products for optimisation by the chosen optimality criteria.

V. Optimising calculations of supply chain parameters must be presented in a form convenient for use. The form of representing results of optimisation must facilitate the adoption and implementation of administrative decisions by managers. The development and application of unique decision-making algorithms are needed where applied supply chain optimisation parameters are one of the key factors [20]. Though it is still an essential element, the algorithm used to develop the optimal solution in supply chains must be flexible, adaptable and confirmable. This will facilitate the implementation of management decisions regarding supply chains.

VI. Optimisation requires the qualified professionals of companies to search for the best logistics decision [21]. This principle and requirement provide scientific and almost reasonable optimisation objectives, including intentional function, optimality criteria, a system of restrictions of the economic-mathematical model of the logistics decision, and modern software. It is not necessary to assume the correct objective definition of optimisation and the effective use of computer programs. This is particularly true for workers who do not posses the necessary knowledge in this area or experience in optimising calculations.

VII. Monitoring supply chains and logistics business processes subject to optimisation. The business processes for which optimisation is carried out must be supported according to goals and developed algorithms. However, this does not exclude their continuous improvement by managing changes and the emergence of more effective software products.

3 Results and discussion

The monitoring of optimised supply chains is supplemented with an assessment and analysis of optimisation costs. The maintenance of and changes to initial optimised parameters, as the improvement of supply chains, demand considerable technology and personnel costs. Also necessary are the assessment of the total costs of optimisation and the comparison of a previous decision with control alternatives. The definition of the impact of the optimisation of technology on the economic indicators of an organisation requires benchmarking. This might relate to crucial indicators of efficiency before technological implementation, the comparison of optimisation results with control indicators and the performance of regular audits of optimised business processes.

A critical place in supply chain management is taken by the optimisation of the arrangements of a warehouse chain in the territory served. The optimisation of a logistics chain includes analysing data and logistics strategy elements for the definition of quantity and delivery volumes, and the arrangement of distribution centres to achieve an optimum balance between the level of service and logistics costs. The optimisation of a chain allows us to increase service quality and achieve significant efficiency in terms of the maintenance costs of a warehouse, the transportation of goods and investment. Growing interest in the optimisation of chains among professional logistics providers has caused significant growth in the software market for optimisation over the last five years. However, many companies mistakenly carrying out optimisation based only on the analysis of data. By paying too much attention to economic-mathematical modelling, companies miss the strategic and practical contexts of optimisation, which may lead to a severe reduction in their client base. The characteristics of the shipment of goods through optimisation software may consider these critical, but less operational factors.

The optimisation possibilities of software have improved considerably over the last five years and now allow us to carry out complex factorial analysis [22]. However, logistics specialists must rely not only on the

modelling instruments of decision-making support, but also on the defining factors of creating a distributive chain. The best approach consists of the optimum combination of these tools that facilitate the economic-mathematical modelling of a distributive chain. This includes practical questions regarding a logistic chain's arrangement and objective statements, and the development of the corresponding strategy.

Initially, it is necessary to consider the shortcomings of the specific optimisation of a chain. It is then possible to offer a modern approach to carrying out similar optimisation. This considers the strategic and practical questions regarding the placement of a logistic chain and their integration with optimisation results. Companies wishing to optimise logistics networks spend most of the time collecting and developing accurate operational estimates of costs to satisfy data software package requirements. Considerable efforts are necessary for processing, analysing and verifying data to accurately understand their general corporate strategy with regard to their impact on the supply chain. Meetings with clients to plan future service parameters of the chain using data serve as secondary sources for analysis. A company can afford or delay optimising a chain or consider potential supply chain harmonisation without optimising the needs of its supply chain participants due to the high probability of the need to purchase assets for the development and optimisation of the chain. This is not less important than the practical strategic objective of achieving logistics chain optimisation.

Support for the adoption of the logistics decision to optimise a network is provided using modern software [18], which gives significant assistance for assessing collected data regarding the quantitative and qualitative parameters of a network and the productive parameters obtained from economic-mathematical modelling.

3.1 Application and implication of the model

In the integrated supply chain management world, the textile companies seek to optimise supply chains and the functional area of logistics, and the business processes of transportation, warehousing and distribution to achieve the maximum results while optimising current costs and resources [23]. In order to optimise economic streams at companies and in supply chains, the managers of foreign textile companies use well-known methods and ways that might include six sigma, economical production, integrated quality control, complicated computer modelling instruments, and the planning of deliveries, the use of modern technologies of management, and other numerous optimisation methods [24].

In the broadest terms, optimisation means balancing several factors to achieve the best overall result. In planning, for example, optimisation means balancing the use of transport and operational costs, i.e. the reserve rate, including customer service. The prices of finished goods and raw materials, outputs or a combination of business processes are balanced to achieve cooperation. In the processing mode, transaction optimisation means using modern software to choose the best alternative processes, such as the routing of shipment or distribution of production [25].

We must, however, take into account the best possible decision that provides the maximum result in each specific situation. This is impractical as its achievement requires high implementation costs. For example, textile companies develop an optimal distributive chain variant. Computer modelling can build an optimum chain of similar distribution on several markets and place the distributor's primary distribution centre on several markets. From a practical point of view, however, a better approach is to implement a decision on only one market. In other words, instead of looking for the ideal decision, it is better to choose the practical decision for each specific situation.

We can add to the central questions of optimising logistics business processes and supply chain management the definition of its purpose, optimality criteria, and the corresponding restrictions regarding time and resources. In strict economic-mathematical terms, optimisation represents the process of searching for parameters, such as economic streams, logistics business processes and supply chain management. By using them, the extreme (minimum or maximum) value of the indicator (vector) chosen by the optimality criteria is achieved [18].

Companies took a huge step forward in data processing automation, deliveries connected with a particular chain and logistics operations. While these innovations reduced costs due to decreased labour skills, their most significant impact is expected in the future. The automation of data processing is an essential subsystem of optimising the supply chain, and allows most textile companies to reduce their costs and increase efficiency significantly. There is an opportunity to reduce costs by 10 to 40% through more effective logistics decisions for many supply chains.

4 Conclusion

Findings

This research suggests that optimising and managing the supply chains of textile producers requires the optimisation of other costs and transport expenses, including optimal placement when establishing new textile enterprises. Thus, the satisfaction of the need for the effective control and management of all logistic chains, i.e., supply, production, transportation and textile production will lead to positive results when penetrating the organisational structure of a new business that is technologically adjacent to an existing production and marketing chain. It was also highlighted that optimisation models must be adequate and correctly illustrate the logistics business processes and functions of supply chain management.

Limitations

This research focused on only one country, i.e., Uzbekistan, and thus might not apply to some other countries. This research deals with well-established models and lacks the latest statistical or mathematical models. More care should be taken to ensure that data regarding logistics, supply chains and business processes are exact, timely and quick due to their use in optimisation models, which results in a significant variation depending on the values of factorial signs and system of restrictions.

Future suggestions

The purposes of optimisation must to be measurable and correspond to the optimality criteria of the actual logistic decision that it has to be reflected in the statement of the related task. For example, the economic-mathematical model of sales optimisation of finished goods can be carried out using the optimality criteria to maximise sales and profit, while minimising used resources and costs. The use of artificial neural networks and artificial intelligence might be applied in the future.

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Analiza okoljskih kriterijev v poročilih o trajnostnem razvoju podjetij v tekstilnem in oblačilnem sektorju

Analysis of Environmental Criteria in Sustainability Reports of Companies in the Textile and Apparel Sector

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Izvleček

Tekstilna panoga je pogosto deležna kritik, ker zelo negativno vpliva na okolje in ker so delovne razmere v njej v državah v razvoju zelo nehumane. Eden od ciljev odgovornega in trajnostnega ravnanja je tudi transparentnost pri poročanju o vplivih podjetja na okolje in izvajanju ukrepov okoljske politike, pri čemer imajo pomembno vlogo poročila o trajnostnem razvoju. V ta namen smo z analizo vsebine proučili poročila o trajnostnem razvoju izbranih podjetij tekstilne in oblačilne panoge s poudarkom na okoljskih kriterijih. Raziskali smo, o katerih okoljskih kriterijih in ukrepih poročajo izbrana podjetja in ali se je zavedanje o okoljskih problemih in o številu ukrepov, ki jih proučevana podjetja povzročajo s svojo dejavnostjo, v opazovanem časovnem obdobju poglobilo. Rezultati so potrdili, da ogljični in vodni odtis v zadnjih letih močno pridobivata na pomenu. V okviru življenjskega cikla oblačil v proučevanih podjetjih najpogosteje namenjajo največjo pozornost ukrepom v fazi proizvodnje in pridobivanja surovin, najmanj pa v fazi uporabe. Vsa so v svojih poročilih navajala zgolj pozitivne informacije, le v majhni meri so bili navedeni tudi neuspehi pri doseganju določenih ciljev trajnostnega razvoja.
Ključne besede: tekstilna panoga, oblačilna industrija, poročila o trajnostnem razvoju, okoljski kriteriji, ogljični odtis

Abstract

The textile industry is often criticized for its enormous negative impact on the environment and non-human working conditions, especially in third-world countries. One of the goals of sustainability measures is the transparency of communications regarding environmental impacts and the policy measures of companies. So-called sustainability reports have become one of the most popular ways to communicate with stakeholders regarding the sustainability efforts of companies. In this paper, we analysed the content of the sustainability reports of textile industry and apparel companies with an emphasis on environmental criteria. We focused on the number of environmental criteria and analysed the measures taken in a given period. The results revealed that, in all cases, more detailed information regarding the carbon and water footprint were reported every year. In general, companies reported the most about measures taken in the production and raw material extraction phases, and the least about the consumption stage. However, mostly positive information about environmental aspects were included in the analysed sustainability reports. Failures regarding sustainable development programmes were rarely mentioned.
Keywords: textile sector, apparel industry, sustainability reports, environmental criteria, carbon footprint

1 Uvod

Trajnostni vidiki so se prebili med pomembnejše vidike v tekstilni industriji [1–4]. Zahteve po zmanjševanju negativnih vplivov na okolje in človeku primernih delovnih razmerah prihajajo od kupcev, konkurence, neprofitnih organizacij in iz državnih ustanov [2, 5]. Kupci v zadnjih letih skupaj z mediji, nevladnimi organizacijami in lokalnimi skupnostmi pritiskajo na podjetja tekstilne in oblačilne industrije, da začnejo delovati v smeri trajnostnega razvoja [6–8]. Samo oblačilna industrija naj bi bila odgovorna za 2–10 odstotkov vseh vplivov na okolje v okoljskih življenjskih ciklih izdelkov v EU [9].

Proizvodnja tekstilnega izdelka zajema veliko proizvodnih faz, ki se pogosto izvajajo v različnih delih sveta. Posledično imajo tekstilna podjetja zapletene globalne dobavne verige [10–11], ki vključujejo veliko različnih faz in udeležencev [12–13]. Še posebno resna so opozorila v povezavi s proizvodnjo oblačil, kjer je za vsako fazo življenjskega cikla mogoče najti skrb zbujajoče podatke bodisi o onesnaževanju glede velike porabe vode, škodljivih kemikalij, fosilnih goriv in nastajanja različnih vrst odpadkov [7, 12, 14–15] bodisi o delovnih razmerah v t. i. državah tretjega sveta [5, 16–17]. Zaradi globalnih in kompleksnih dobavnih verig oblačila prepotujejo velike razdalje od začetka svojega življenjskega cikla do končnih uporabnikov, kar znatno povečuje njihov ogljični odtis [10]. K resnim okoljskim vplivom pripomore tudi prodobivanje bombaža. Po nekaterih podatkih bombaž zaseda 2,4 odstotka celotne obdelovalne površine na svetu, povečanje števila nasadov pa zmanjšuje biotsko raznovrstnost narave [18–19]. Za pridelovanje bombaža se porabi kar šest odstotkov vseh pesticidov in 16 odstotkov vseh insekticidov na svetu [20]. Tekstilna industrija je tudi sicer velik porabnik kemikalij, kar je tudi eden osrednjih virov onesnaženja vode v tekstilni proizvodnji, ki je posebej evidenten v državah z nizkimi okoljevarstvenimi standardi in zakonodajo [21–22].

Ob tem je oblačilna industrija pod velikimi pritiski tudi zato, ker povzroča velikanske količine trdnih odpadkov, ko se oblačila zavržejo [23]. To je posledica poslovnega modela, ki temelji na pretiranem potrošništvu oziroma na t. i. trendu hitre mode, kar pomeni, da oblačila čim hitreje pridejo iz proizvodnje na trg, in to po čim nižji ceni, ob nenehnem spodbujanju novih nakupov. V ta namen se proizvodnja preseli v države z nižjimi stroški dela. Tako so oblačila cenejša in jih odjemalci lahko kupijo v večjih količinah [24]. Vendar velika količina kupljenih oblačil slabše kakovosti posledično povzroči, da jih uporabniki hitreje zavržejo [14]. Kot navaja Draper s soavtorji [10], so prav razpršene in nepregledne dobavne verige ter hitra moda glavni problemi te panoge. Zaradi neodvisnih raziskav in raznih škandalov, v katere so bila vpletena velika podjetja oblačilne industrije, se zaupanje javnosti v ta podjetja zmanjšuje, kar zmanjšuje ugled celotne panoge [4]. Ne glede na kompleksnost problema tudi v tej panogi že obstajajo premiki glede implementacije trajnostnih vidikov [25] in rast proaktivnih praks [26].

Zato je postala eden od ciljev trajnostnega razvoja tudi transparentnost pri poročanju o vplivih podjetja na okolje in izvajanju ukrepov okoljske politike [4, 6, 8, 11], kar vključuje tudi pripravo in objavljanje poročil o trajnostnem razvoju. Le-ta postajajo v številnih panogah čedalje pomembnejša za komuniciranje z javnostjo o aktivnostih podjetja, ki pripomorejo k trajnostnemu razvoju [27–31]. Poročila o trajnostnem razvoju omogočajo podjetjem, da predstavijo svoje aktivnosti na področju okolja in družbe, hkrati pa javnosti omogočajo vpogled v poslovanje podjetja in način prispevanja k trajnostnemu razvoju. Poročanje o trajnostnem razvoju je v zadnjem desetletju v izrazitem porastu v različnih panogah [27, 32]. To lahko pripišemo tudi povečani uporabi smernic poročanja Global Reporting Initiative (GRI), ki so priporočila za poročanje o ekonomskih, socialnih in okoljskih vidikih poslovanja podjetja. Upoštevanje smernic poročanja GRI poveča primerljivost, kakovost in poenotenost informacij podjetij o njihovih (pozitivnih in negativnih) prispevkih k trajnostnemu razvoju [29, 33]. Čeprav število podjetij s področja tekstilne panoge v svetu, ki se odločajo, da bodo delila trajnostne podatke z javnostjo, raste, pa je delež tovrstnih podjetij v določenih državah še vedno razmeroma majhen [34].

Pregled spletnih bibliografskih baz je pokazal, da je število raziskav, v katerih je bilo sistematično proučeno, kateri trajnostni indikatorji so najpogosteje predstavljeni na področju tekstilne panoge (in na kakšen način), še vedno zelo omejeno tudi v mednarodnem merilu, na kar opozarjajo avtorji sami [26, 35]. Čeprav so dosedanje objave zagotovo pripomogle k boljši preglednosti in razumevanju trendov ter pristopov podjetij v tekstilni panogi glede trajnostnega poročanja, pa je še vedno na voljo premalo podatkov za razumevanje celovite slike v kompleksnih dobavnih verigah te panoge in potrošniških navadah. Na

splošno je takšno poročanje ponavadi kombinacija kvantitativnih in kvalitativnih kazalnikov, bodisi na spletnih straneh [35] bodisi v okviru trajnostnih poročil [11, 36], med katerimi prevladujejo okoljski vidiki in opisi delovnih razmer [26]. Več avtorjev poroča, da se trajnostni indikatorji najpogosteje nanašajo na ukrepe v dobavnih verigah, manj pa na poročanje o poslovnih inovacijah in spreminjanju potrošniških navad [11, 37]. Ob tem obstajajo še številna odprta vprašanja glede standardizacije, verifikacije in verodostojnosti trajnostnih poročil ter s tem njihove medsebojne primerljivosti, kar sicer ni značilno le za tekstilno oz. oblačilno panogo [38]. To potrjujejo izsledki študij, o katerih poročajo Kozlowski in soavtorji [37] ter Garcia-Torres in soavtorji [39], ki nakazujejo na vrzeli v konsistentnosti in verodostojnosti pri trajnostnem poročanju podjetij tekstilne panoge v svetu.

Zaradi omenjenih razlogov postajajo analize objavljenih okoljskih in trajnostnih poročil v zadnjih letih pomembno področje raziskav trendov trajnostnega razvoja [30, 36, 40–42]. Z njimi želimo pridobiti informacije o strukturi poročil, načinih komuniciranja, izbranih trajnostnih kriterijih, razvojnih dosežkih in razlikah med panogami. Spoznanja iz raziskav o trajnostnem poročanju so pomembna tudi z vidika objektivnosti poročanja v prihodnje, saj so objavljeni podatki žal velikokrat zavajajoči in nepopolni [43, 44].

Namen raziskave je opraviti primerjalno analizo poročil o trajnostnem razvoju izbranih podjetij tekstilne panoge in ugotoviti, o katerih okoljevarstvenih kriterijih in izvedenih okoljskih ukrepih proučevana podjetja poročajo. Ob tem želimo raziskati, ali se trajnostno poročanje in s tem zavedanje podjetij tekstilne panoge o okoljskih problemih, ki jih povzročajo s svojo dejavnostjo, povečuje ali ne. S tem želimo prispevati k spoznavanju dinamike trajnostnega poročanja v določenih časovnih obdobjih, kjer so podatki še zelo pomanjkljivi.

Znanstvenih prispevkov, ki bi v slovenski prostor prinašali spoznanja o pomenu, značilnostih in pomanjkljivostih trajnostnega poročanja, za podjetja ni veliko. Rezultati raziskave so namenjeni podjetjem slovenske tekstilne panoge, a tudi drugih panog slovenskega gospodarstva v zvezi s trajnostnim poročanjem. Disipacija tovrstnih informacij in spoznanj je pomembna in nujna za implementacijo objektivnega trajnostnega poročanja tako v slovenski industrijski kot tudi trgovinski dejavnosti. Razumevanje problema vplivov na okolje, ki jih povzročajo (velika) podjetja, in razumevanje pravilnega informiranja in poročanja o tem je pomembno tudi za vzpostavitev trajnostnih politik malih in srednje velikih podjetij (ki so pogosto dobavitelji velikim podjetjem), saj jim velika podjetja postavljajo določene zahteve, ki jih morajo izpolniti. Brez tega razumevanja bodo tudi mala in srednje velika podjetja v vseh gospodarskih panogah, vključno s tekstilno, v prihodnosti zagotovo manj konkurenčna.

2 Metodologija raziskave

2.1 Opis vzorca raziskave

Kriterij za izbor podjetij za raziskavo je bilo javno dostopno poročilo o trajnostnem razvoju (angl. *Sustainability report*), objavljeno na spletni strani podjetja. V raziskavo smo vključili trajnostna poročila naslednjih podjetij: Adidas Group, C&A, Gap Inc., H&M in Nike Inc. V poročilih teh podjetij so informacije za celotno tekstilno panogo, od pridobivanja tekstilnih surovin do plemenitenja tekstilij. Podatki se nanašajo na dobaviteljska podjetja, ki jih uvrščamo v ožji pomen tekstilne panoge, saj se ukvarjajo s proizvodnjo tekstilnih materialov kot končnih tekstilnih izdelkov. Zavedamo se, da gre za specifičen vzorec podjetij tako glede izdelkov, ki jih ponujajo na trgu (oblačila, obutev), kot glede na poslovne modele in korporacijsko organiziranost. Uporabili smo tiste informacije iz poročil, ki se nanašajo na oblačilne izdelke, ki jih sicer vsa ta podjetja ponujajo na trgu. Dejstvo pa je, da imajo tekstilni izdelki v obliki oblačil za izbrana podjetja pomemben tržni delež in da so podjetja vpeta v vse faze dobavne verige, vključno s tekstilno proizvodnjo (tekstilnih materialov in ploskovnih tekstilnih izdelkov), na katero močno vplivajo ter o zbranih okoljskih podatkih svojih dobaviteljev iz tekstilne panoge tudi poročajo. Njihova trajnostna poročila so informacijska baza, v kateri so na enem mestu zbrani podatki o njihovih dobaviteljih v celotni tekstilni verigi, vključno s pridobivanjem oz. proizvodnjo tekstilnih materialov, prej, ploskovnih tekstilnih izdelkov s plemenitenjem in konfekcioniranjem.

Izbor podjetij dodatno utemeljujemo z naslednjim:

(1) Velika podjetja imajo na splošno laže dostopna in obsežnejša poročila o trajnostnem razvoju kot mala in srednje velika.

(2) Imajo veliko tržno moč in s tem veliko družbeno odgovornost. Velika globalna podjetja (lastniki mod-

nih znamk) imajo največji vpliv na to, kakšne izdelke bodo izdelovali in pod kakšnimi pogoji.
(3) So pod večjim pritiskom javnosti.
(4) Močno vplivajo na spremembe v dobavnih verigah zaradi zahtev, ki jih lahko postavljajo dobaviteljem. So tista, ki pogosto sprožijo trajnostne spremembe in v to prisilijo tudi mala in srednje velika podjetja, ki so njihovi dobavitelji ali odjemalci.
(5) Trendom, ki so trenutno prepoznani za velika podjetja, se pozneje marsikdaj pridružijo tudi mala in srednje velika podjetja. Trajnostne zahteve, ki se tako prenašajo z večjih na manjša podjetja, so gibalo sprememb v smeri trajnostnega razvoja v panogi.
(6) Razpolagajo z več kadrovskimi, raziskovalnimi in finančnimi potenciali, ki jih lahko vložijo v ekoinovacije.

2.2 Analiza poročil in uporabljene metode

Raziskava temelji na kvalitativni analizi poročil, ki so izrecno ocenjena kot trajnostna (angl. sustainability report). V ta namen smo marca leta 2019 pregledali spletne strani izbranih podjetij. Za proučevanje napredka pri trajnostnem poročanju smo se omejili na poročila, izdana v zadnjih treh do petih letih za vsako izbrano podjetje. Za vsako od izbranih podjetij smo pregledali tri najnovejša dostopna poročila o trajnostnem razvoju, pri čemer so določena poročila izdana za eno leto, nekatera pa za dve leti skupaj (preglednica 1).
Pri trajnostnih kriterijih (okoljski, socialni, ekonomski) se v raziskavi omejujemo na okoljske kriterije: izvor surovin tekstilnih materialov, uporaba kemikalij, raba energije, vodni odtis, ogljični odtis in trdni odpadki. Kot navaja več avtorjev, prav ti vidiki pomenijo področja, ki največ prispevajo k okoljskim obremenitvam v celotnem življenjskem ciklu tekstilnih izdelkov [14, 19, 21]. Kot posebej pomembne za tekstilno industrijo jih omenjajo tudi poročila Evropske unije in nevladne organizacije [1–2, 14].
Za proučevanje poročil o trajnostnem razvoju izbranih podjetij smo uporabili metodologijo, ki je znana kot ‚analiza vsebine' (angl. Content analysis), katere metodološki raziskovalni standard je delo avtorja Klausa Krippendorfa [60] in je pogosto uporabljena za raziskave trajnostnih poročil tekstilnih oziroma oblačilnih podjetij [26, 37, 39]. Krippendorf opredeljuje analizo vsebine kot raziskovalno metodo za pridobitev ponovljivih in verodostojnih tekstovnih informacij glede na kontekst njihove uporabe, in sicer za določanje podobnosti in razlik med proučevanimi tekstovnimi podatki, pa tudi za iskanje povezav med njimi. Metodologija analize vsebine pomaga zmanjšati količino zbranih podatkov in jih razvrstiti v določene skupine. Analiza vsebine poteka tako, da najprej zberemo relevantne podatke, jih sistematično pregledamo z uporabo izbranih kriterijev ter določimo podobnosti in razlike med proučevanimi podatki, pa tudi povezave med njimi [61]. Analizo vsebine smo uporabili, ker je široko uporabljan metodološki okvir pri raziskovanju trajnostnih in sorodnih poročil [62–65].
Pri analizi besedil smo bili pozorni na to, da izbrani kriteriji niso bili zgolj omenjeni v besedilu, ampak da so bili zanje podani dejanski opisni in številski podatki. Za vsak okoljski kriterij smo preverili in analizirali kvalitativne in kvantitativne podatke. Pristop, ki smo ga uporabili, ne razvršča pojmov glede na njihovo relativno pomembnost, temveč glede na njihovo pogostnost. Ob tem smo dodatno proučili, kako izbrana podjetja v svojih poročilih poročajo o ukrepih v različnih fazah okoljskega življenjskega cikla. Prav tako smo proučili, kako podjetja v svojih poročilih poročajo o ukrepih za zmanjševanje vplivov na okolje.

Preglednica 1: Seznam upoštevanih poročil o trajnostnem razvoju tekstilnih podjetij
Table 1: List of considered sustainability reports of selected companies

Podjetje/ *Company*	Leto izdaje prvega pregledanega poročila/ *Year of publication of first examined report*	Leto izdaje drugega pregledanega poročila/ *Year of publication of second examined report*	Leto izdaje tretjega pregledanega poročila/ *Year of publication of third examined report*	Viri/ *References*
Adidas Group	2014	2015	2016	45–47
C&A	2014	2015	2017	48–50
Gap Inc.	2013–2014	2015–2016	2017	51–53
H&M	2015	2016	2017	54–56
Nike Inc.	2012–2013	2014–2015	2016–2017	57–59

3 Rezultati in razprava

V preglednicah 2 in 3 so prikazani rezultati analize vsebine poročil o trajnostnem razvoju za izbrana podjetja (preglednica 1). Treba je poudariti, da v okviru uporabljene metodologije v preglednicah 2 in 3 navedene ukrepe podjetij predstavljamo v enaki terminološki obliki, kot so zapisani v poročilih, se pravi z dejanskimi izrazi, kot so jih navajala podjetja. Posledično to pomeni, da se v poročilih lahko pojavljajo drugačni oz. sorodni termini za iste ukrepe. V tabeli 2 so navedeni ukrepi za vsak proučevani okoljski kriterij posebej in kvantitativne vrednosti zmanjševanja vplivov na okolje. Velikosti zmanjšanja vplivov se razlikujejo glede na posamezni okoljski vpliv oziroma glede na fazo okoljskega življenjskega cikla izdelkov. Opazne razlike so tudi med posameznimi podjetji. Videti je, da podjetja o nekaterih ukrepih pogosteje poročajo. Na primer, vsa proučevana podjetja poročajo o zmanjševanju porabe vode v proizvodnji. Prav tako skoraj vsa poročajo o ukrepih za zmanjševanje izpustov toplogrednih plinov (kar je povezano z ogljičnim odtisom). V vseh pregledanih poročilih je bilo opaziti velik poudarek na izboru surovin, vendar nikjer nismo zasledili konkretnih podatkov o ravnanju z vodo in uporabi kemikalij pri pridelavi bombaža, kar je do določene mere skladno s spoznanji nekaterih drugih avtorjev [26]. Vsa izbrana podjetja poročajo o rabi vode v proizvodnih procesih dobaviteljev, ne poročajo pa o ravnanju z odpadno vodo. Prav tako vsa podjetja omenjajo, da iz proizvodnih procesov izločajo zdravju nevarne kemikalije, nismo pa zasledili podatkov o ravnanju z uporabljenimi kemikalijami, ki se marsikdaj izpuščajo v bližnje reke, na kar opozarjajo nevladne organizacije [3]. Tudi Kozlowski in soavtorji [37] poročajo, da se okoljski kazalniki v trajnostnih poročilih tekstilnih podjetij sicer nanašajo na vse faze življenjskega cikla, vendar so ukrepi neenakomerno porazdeljeni po posameznih fazah. S primerjavo teh spoznanj z našimi se zdi, da dajejo podjetja v tem trenutku večji poudarek tistim ukrepom, ki so povezani z največkrat jim očitanimi vplivi na okolje v širši javnosti in v medijih. Za dejansko primerljivost podatkov glede zmanjševanja vplivov na okolje in s tem primerljivostjo med podjetji samimi bi bile potrebne enotne in jasne mednarodno standardizirane zahteve glede trajnostnega poročanja. Ob tem želimo poudariti, da v skladu z namenom in cilji raziskave proučujemo, kaj in kako podjetja poročajo, ne pa tudi, s čim in na kakšen način te ukrepe dosegajo (npr. s katerimi procesnimi, izdelčnimi ali organizacijskimi inovacijami).

Dinamika zmanjševanja vplivov izbranih podjetij na okolje je dodatno prikazana v tabeli 3, v kateri so navedeni ukrepi za vsako posamezno fazo okoljskega življenjskega cikla posebej, in sicer za primer tistih podjetij, ki so v svojih poročilih dejansko prikazala napredek z navajanjem konkretnih številskih podatkov in niso določenega kriterija le tekstovno omenjala. Prikazana je razlika med prvim in zadnjim proučevanim letom poročanja, vendar le za tista podjetja, ki so navajala konkretne številske podatke (torej niso določenega pojma oziroma okoljskega ukrepa samo omenjala v besedilu). Videti je, da se največ ukrepov nanaša na fazo proizvodnje in na fazo pridobivanja surovin. Bistveno manj pogosto podjetja poročajo o ukrepih glede faze uporabe, ravnanja s tekstilnimi odpadki po uporabi in o transportu, čeprav so ti trije prav tako pomembni.

Faza uporabe se nanaša na vzdrževanje tekstilnih izdelkov (pranje, sušenje in likanje). Večkrat se je potrdilo, da je ta faza življenjskega cikla lahko celo med okoljsko najbolj obremenjujočimi [66]. Res je, da je v tem primeru vpliv na okolje (raba energije, vode in detergentov) v največji meri odvisen od potrošnikov in aparatov, ki jih uporabljajo [1], vendar bi jih izdelovalci lahko pogosteje opozarjali, saj imajo za to na voljo različne možnosti (spletne strani, družbena omrežja, etikete). Potrošniki imajo izjemno pomembno vlogo pri spremembah v smeri trajnostnega razvoja, saj s svojo izbiro o nakupu oblačil in pritiski na velika podjetja tekstilne industrije silijo le-ta v bolj odgovorne odločitve. Le Gap Inc. in H&M sta poudarila, da kupce izobražujejo o načinu pranja in sušenja oblačil, da bi čim bolj zmanjšali vpliv na okolje v omenjeni fazi.

V poročilih je tudi relativno malo podatkov o fazi ravnanja s tekstilnimi odpadki, kar lahko pripišemo dejstvu, da so metode recikliranja oblačil še vedno v fazi razvoja. Vsa izbrana podjetja so poročala o tem, da so (vsaj za določen čas) že organizirala programe zbiranja rabljenih oblačil v trgovinah, le dve od izbranih podjetij, H&M in C&A, pa sta navedli, da sodelujeta s podjetjem I:CO, ki se ukvarja s sortiranjem zbranih oblačil in njihovo ponovno uporabo ali recikliranjem [67]. Najpomembnejša intervencija za izboljšanje položaja tekstilne industrije bi zagotovo bila zmanjšana poraba oblačil oziroma izdelovanje kakovostnih oblačil z dolgo življenjsko dobo ob hkratnem upoštevanju okoljskih in socialnih problemov [68], kar pa bi resno poseglo v poslovni model hitre mode. Vprašanje je, ali si izbrana podjetja želijo posegati vanj, čeprav je prav hitra moda danes eden

Preglednica 2: Poročanje o okoljskih kriterijih in ukrepih v trajnostnih poročilih
Table 2: Reporting on environmental criteria and measures in sustainability reports

Okoljski vplivi/ *Environmental impacts*	Ukrepi proučevanih podjetij, za katere so v poročilih na voljo konkretni številski podatki/ *Measures taken, reported by quantitative data*	Število podjetij, ki so poročala/ *Number of reporting companies*	Napredek v obravnavanem obdobju/ *Improvements achieved in examined period of time*
Izvor tekstilnih materialov/*Source of textile materials*	Uporaba trajnostno pridobljenega bombaža/ *Use of sustainable cotton*	5	Od 2014. do 2016. povečanje za 38 % [45-47]./ *From 2014 to 2016, increase by 38% [45–47]*
			Leta 2017 10–krat več trajnostnega bombaža kot leto prej [52-53]./ *In 2017, 10 times more sustainable cotton than the year before [52–53]*
			Leta 2015 40 %, leta 2017 67 % [48-50]./ *In 2015, 40%; in 2017, 67% [48–50]*
			Leta 2015 34 %, leta 2017 59 % [54-56]./ *In 2015, 34%; in 2017, 59% [54–56]*
			Leta 2015 24,1 %, leta 2017 54,1% [58-59]./ *In 2015 24.1%; in 2017, 54.1% [58–59]*
	Uporaba recikliranega poliestra/ *Use of recycled polyester*	2	Leta 2017 1,7 % (v prejšnjih poročilih ne omenjajo številskih podatkov) [53]./ *In 2017, 1.7% (they do not mention numerical data in previous reports) [53]*
			Leta 2015 uporabili 31.220 t, leta 2017 33.265 t [58-59]./ *In 2015, 31,220 t were used; in 2017, 33,265 t [58–59]*
	Uporaba trajnostno pridobljenih materialov/*Use of sustainable-sourced materials*	2	Leta 2015 20 %, leta 2017 35 % [54-56]./ *In 2015, 20%; in 2017, 35% [54–56]*
			Leta 2015 19 %, leta 2017 29,6 % [58-59]./ *In 2015 19%; in 2017, 29.6% [58–59]*
Uporaba kemikalij/*Use of chemicals*	Oblačila brez perfluoriranih spojin/*Perfluorocarbon-free finished products*	1	Leta 2014 90 % oblačil, 2016 96 % [45-47]./ *In 2014, 90% of clothing, in 2016, 96% [45–47]*
Raba energije/ *Energy consumption*	Raba energije iz obnovljivih virov v lastnih procesih/*Consumption of renewable and sustainable energy in own processes*	2	Leta 2015 78 %, v letu 2017 96 % [54-56]./ *In 2015, 78%; in 2017, 96% [54–56]*
			Leta 2015 14 %, leta 2017 22 % [57-59]./ *In 2015, 14%; in 2017, 22% [57–59]*

Okoljski vplivi/ *Environmental impacts*	Ukrepi proučevanih podjetij, za katere so v poročilih na voljo konkretni številski podatki/ *Measures taken, reported by quantitative data*	Število podjetij, ki so poročala/ *Number of reporting companies*	Napredek v obravnavanem obdobju/ *Improvements achieved in examined period of time*
	Zmanjšanje rabe energije v lastnih procesih/*Energy consumption reduction in own processes*	1	Od leta 2007 do 2017. zmanjšanje za 9 % [54-56]./ *9% reduction from 2007 to 2017 [54–56]*
	Zmanjšanje rabe energije v proizvodnji pri dobaviteljih/ *Energy consumption reduction in own processes*	2	Od leta 2015 do 2017. zmanjšali porabo za pribl. 1 kW za kg oblačil v fazi barvanja in konfekcioniranja [58-59]./*From 2015 to 2017, energy consumption reduction by approx. 1 kW per kg of clothing in the dyeing and finishing processes [58–59]*
			Leta 2015 zmanjšali porabo za 30 milijonov kWh, leta 2017 za 98 milijonov kWh [54-56]./*In 2015, they reduced consumption by 30 million kWh, in 2017 by 98 million kWh [54–56]*
Vodni odtis/ *Water footprint*	Zmanjšanje porabe vode/*Water consumption reduction*	5	Poraba 121 l/kg oblačil leta 2014 in 95 l/kg oblačil leta 2016 [45-47]./ *Consumption of 121 l/kg of clothing in 2014 and 95 l/kg of clothing in 2016 [45–47]*
			Od leta 2016 do 2017. zmanjšanje za 14 % [50]./ *From 2016 to 2017, a decrease of 14% [50]*
			Od leta 2014 do 2016. prihranili 3,3 mrd l; leta 2017 prihranili 2,4 mrd l [51-53]./ *From 2014 to 2016, they saved 3.3 billion l; in 2017, they saved 2.4 billion l [51–53]*
			V letu 2016 prihranili 2,3 milijona m^3, v letu 2017 pa 7,82 milijona m^3 [55-56]. Leta 2017 za 55 % kavbojk med proizvodnjo ni bilo porabljeno več kot 35 l vode na kos, leta 2015 takšnih 50 % kavbojk [54-56]./ *Saved 2.3 million m^3 in 2016 and 7.82 million m^3 in 2017 [55–56]. In 2017, 55% of jeans did not consume more than 35 l of water per piece during production, in 2015 50% of such jeans [54–56]*
			Od leta 2015 do 2017. zmanjšali porabo za skoraj 10 l na kg oblačil v fazi barvanja in konfekcioniranja [58-59]./ *From 2015 to 2017, reduction of nearly 10 l per kg of clothing in the dyeing and finishing processes [58–59]*

Okoljski vplivi/ *Environmental impacts*	Ukrepi proučevanih podjetij, za katere so v poročilih na voljo konkretni številski podatki/ *Measures taken, reported by quantitative data*	Število podjetij, ki so poročala/ *Number of reporting companies*	Napredek v obravnavanem obdobju/ *Improvements achieved in examined period of time*
Ogljični odtis/ *Carbon footprint*	Zmanjšanje izpustov toplogrednih plinov v lastnih procesih/ *Greenhouse gases emissions reduction in own processes*	3	Od 2015. do 2016 zmanjšanje za 11 % [46-47]./ *From 2015 to 2016, a decrease of 11% [46–47]*
			Od 2008. do 2014. zmanjšanje za 33 % [51]./ *From 2008 to 2014, a decrease of 33% [51]*
			Od leta 2016 do 2017. zmanjšanje za 21 % [55-56]./ *From 2016 to 2017, a decrease of 21% [55–56]*
	Zmanjšanje izpustov toplogrednih plinov pri proizvodnji/ *Greenhouse gases emissions reduction in suppliers' manufacturing processes*	3	Od leta 2016 do 2017. zmanjšanje za 16 % [50]./ *From 2016 to 2017, a decrease of 16% [50]*
			Od leta 2015 do 2016. zmanjšanje za 47 % [54-55]./ *From 2015 to 2016, a decrease of 47% [54–55]*
			Od 2015 do 2017. zmanjšanje s 4,78 na 4,55 kg CO_{2e}/kg oblačil pri barvanju in konfekcioniranju [58-59]./ *From 2015 to 2017, reduction from 4.78 to 4.55 kg CO_{2e}/kg of clothing in dyeing and finishing processes [58–59]*
Trdni odpadki/ *Solid wastes*	Zmanjšanje količine odpadkov v lastnih procesih/*Waste reduction in own operations*	1	Od 2015. do 2016. zmanjšanje za 28 % [46-47]./ *From 2015 to 2016, a decrease of 28% [46–47]*
	Možnost vračanja rabljenih oblačil/*Garment collecting initiative for reuse and recycling*	1	Od 2013. do 2017. so zbrali 57.000 t oblačil [54-56]./ *From 2013 to 2017, 57,000 tons of clothing were collected [54–56]*

Preglednica 3: Poročanje o okoljskih kriterijih in ukrepih za posamezno fazo življenjskega cikla izdelka v trajnostnih poročilih
Table 3: Reporting on environmental criteria and measures in sustainability reports for different life cycle phases

Faza življenjskega cikla/ *Life cycle phase*	Ukrepi podjetij/ *Measures taken by companies*	Število podjetij, ki so poročala v prvem proučevanem poročilu/ *Number of companies that reported in the first examined report*	Število podjetij, ki so poročala v zadnjem proučevanem poročilu/ *Number of companies that reported in the last examined report*
Pridobivanje surovin/ *Raw materials extraction*	Uporaba trajnostno pridobljenega bombaža/*Use of sustainable cotton*	3	5
	Uporaba recikliranega poliestra/ *Use of recycled polyester*	1	2
	Uporaba trajnostno pridobljenih materialov/*Use of sustainable-sourced materials*	1	2
Proizvodnja/ *Production*	Oblačila brez perfluoriranih spojin/ *Perfluorocarbon-free finished products*	1	1
	Zmanjšanje porabe energije v proizvodnji/*Energy consumption reduction in suppliers' manufacturing processes*	0	2
	Zmanjšanje porabe vode v proizvodnji/*Water consumption reduction in suppliers' manufacturing processes*	4	5
	Zmanjšanje izpustov toplogrednih plinov v proizvodnji/*Greenhouse gases emissions reduction within the supply chain*	0	3
Transport/ *Transport*	Vsi prevozniki, s katerimi sodelujejo, so vpisani v bazo Clean Shipping Index, s čimer pripomorejo k zmanjšanju izpustov/*Transport service providers must follow the Clean Shipping Project requirements for emissions reduction*	1	1
Uporaba/ *Use*	Ozaveščanje kupcev o načinu pranja in sušenja oblačil z namenom prihraniti vodo v fazi uporabe/ *Actions that encourage conscious garment care by customers*	0	1
Ravnanje z odpadki/ *Solid wastes treatment*	Možnost vračanja rabljenih oblačil/ *Garment collecting initiative for reuse and recycling*	1	1

glavnih vzrokov obremenjevanja okolja v tekstilni panogi [14, 69].

Čeprav so vsa proučevana podjetja v poročanje zajela svojo dobavno verigo, pa so pri uvajanju izboljšav za rabo energije oz. poročanju o tem še vedno v veliki meri osredotočena na svoje lastne poslovne procese, kamor štejemo prodajalne, distribucijske centre in upravo, čeprav le-ti po njihovih ocenah pomenijo v povprečju zgolj okrog deset odstotkov vseh vplivov podjetja na okolje. Prav vsa proučevana podjetja so zavezana k ciljem prenehanja izpuščanja zdravju nevarnih kemikalij v okolje

do leta 2020 (angl. Zero Discharge of Hazardous Chemicals), kar zahteva sistemske spremembe v celotni dobavni verigi podjetij [3]. To pomeni, da bi velika tekstilna podjetja morala prevzeti odgovornost in pritisniti na svoje dobavitelje, da izvedejo spremembe v smeri trajnostnega razvoja. Skrb zbujajoče je, da je hkrati s poročili o trajnostnem razvoju, ki smo jih proučevali, izšlo veliko poročil neodvisnih institucij, ki še vedno opozarjajo na številne nepravilnosti v povezavi s tekstilno panogo (glej na primer [21, 70–73]), v vsakem od tovrstnih poročil pa je omenjeno vsaj eno od podjetij, ki smo jih zajeli v vzorec raziskave.

O tovrstnih poročilih pa je nujno tudi kritično razmisliti. Vsa proučevana podjetja v svojih poročilih o trajnostnem razvoju sicer omenjajo najbolj pereče probleme tekstilne panoge in določene ukrepe, vendar redkeje poročajo o konkretnem napredku v določenem obdobju. Prav tako vsa proučevana podjetja v svojih poročilih v veliki meri navajajo zgolj pozitivne informacije. Četudi so bila trajnostna poročila v vseh primerih pripravljena na podlagi smernic, ki jih je razvila organizacija Global Reporting Initiative [29], še vedno nimamo mednarodnega standarda za trajnostno poročanje, zato se podatki in načini poročanja v poročilih precej razlikujejo, saj so odločitve prepuščene podjetjem. Roca in Searcy [62] sta na primeru 94 kanadskih podjetij iz različnih panog ugotovila, da le-ta v svojih poročilih uporabljajo kar 585 različnih okoljskih indikatorjev, kar zagotovo ne pripomore k transparentnosti poročanja. O širokem naboru uporabljenih okoljskih kazalnikov za primer trajnostnih poročil v tekstilni panogi poročajo tudi Kozlowski in soavtorji [37] ter Saygili in soavtorji [34]. Zaradi te težave so Garcia-Torres in soavtorji [39] predlagali lasten način vrednotenja okoljskih oz. trajnostnih kriterijev za podjetja, ki so povezana s problemom hitre mode.

Zavedamo se, da so poleg okoljskih vidikov trajnostnega razvoja izjemno pomembni tudi socialni, med katere uvrščamo pošteno plačilo, otroško delo, pravico do združevanja delavcev, izobraževanje zaposlenih, diskriminacijo žensk, diskriminacijo ras, prisilno delo in druge [16, 29, 74]. Čeprav smo v prispevku podrobneje osredotočeni na okoljske kriterije trajnostnega poročanja, smo v ločeni razširjeni študiji proučevali tudi socialne [74]. Ugotovili smo, da večina proučevanih podjetij v svojih poročilih o trajnostnem razvoju po obsegu vsaj polovico poročila namenja tudi socialnim vidikom, med katerimi izstopa vidik ‚delovne razmere‘‘, ki ga proučevana podjetja navajajo v okviru celotne dobavne verige, kar prav tako lahko pripišemo čedalje hujšemu pritisku javnosti [8, 72]. Podrobnejši pregled socialnih kriterijev v trajnostnih poročilih presega namen in cilje tega članka in je podrobneje proučen in opisan v [74].

Tovrstna poročila sicer kažejo, da se podjetje ukvarja s trajnostnim razvojem, vendar pa je tovrstne informacije treba sprejemati tudi z določeno mero pazljivosti, saj je poročanje o trajnostnem razvoju v glavnem usmerjeno k poudarjanju pozitivnih informacij in prikrivanju negativnih [29]. Tako so poročila o trajnostnem razvoju še vedno vse prepogosto namenjena zgolj dobri javni podobi [62, 64, 75–76]. Tudi raziskava Flash Eurobarometer [77] je pokazala, da večina prebivalcev Evropske unije ne zaupa povsem poročilom podjetij, v katerih le-ta objavljajo svoje dejavnosti na področju varovanja okolja. Za pripravo trajnostnih poročil so namreč potrebni verodostojni podatki, pri čemer so za tekstilno panogo (sploh za segment oblačil in hitre mode) dodaten problem dobavne verige in potrošniške navade oziroma razvade. Panoga je razdrobljena na več različnih maloprodajnih segmentov (športna oblačila, luksuzna oblačila, hitra moda ipd.), zato je tudi pojem ‚trajnosti‘ v tej panogi težko enoznačno opredeliti. Tako je implementacija pojma ‚trajnosti‘ še vedno predmet razprav, saj obstajajo specifične zahteve med posameznimi segmenti.

S poročili o trajnostnem razvoju velika podjetja vplivajo na razvoj celotne panoge. Razumevanje celovitih vplivov tekstilne panoge na okolje, ki jih povzročajo velika podjetja na eni strani in razumevanje pravilnega informiranja oz. poročanja na drugi, je pomembno tudi za vzpostavitev trajnostnih politik malih in srednje velikih podjetij (ki so pogosto dobavitelji velikim podjetjem), saj jim velika podjetja postavljajo določene zahteve. Mala in srednje velika podjetja pa s tovrstnimi poročili pridobijo veliko koristnih informacij o trendih v tekstilni panogi, ki jim bodo morala slediti [14]. Tudi prodajalci tekstilnih izdelkov z njimi pridobijo informacije o oblačilih, ki jih prodajajo, kar jim omogoča lažjo komunikacijo z njihovimi kupci in možnost diverzifikacije izdelkov, dobijo pa lahko tudi nove ideje za promocijo. Zato so izsledki raziskave koristni tudi za slovenske modne oblikovalce. Kot navajajo avtorji poročila Fashion at the Cross Roads [68], manjša podjetja v zadnjem času marsikje že vodijo v pozitivnih spremembah tekstilne panoge v smeri trajnostnih zahtev.

4 Sklep

Ker postajajo izzivi trajnostnega razvoja izjemno pomembni za podjetja tekstilne panoge, je pomembno pridobiti čim več verodostojnih informacij za poročanje oz. komuniciranje z javnostjo in deležniki. To ni le značilnost tekstilne panoge, temveč tudi drugih, saj postaja poročanje o trajnostnem razvoju v gospodarstvu čedalje pomembnejše. Z raziskavo smo želeli ugotoviti, kako izbrana podjetja tekstilne industrije v svojih poročilih o trajnostnem razvoju poročajo o izbranih okoljskih kriterijih. Rezultati nakazujejo, da je v proučevanih primerih viden napredek v smeri upoštevanja okoljskih vidikov v obravnavanih obdobjih, saj v svoja poročila o trajnostnem razvoju vsako leto vključujejo več konkretnih podatkov. Ugotovili smo, da čedalje večji pomen pridobivata ogljični in vodni odtis. V okviru življenjskega cikla oblačil se v proučevanih podjetjih najpogosteje osredotočajo na ukrepe v fazi proizvodnje in pridobivanja surovin, najmanj pa v fazi uporabe. Vsa proučevana podjetja so v svojih poročilih navajala zgolj pozitivne informacije, le v majhni meri so bili izpostavljeni tudi neuspehi pri doseganju določenih ciljev trajnostnega razvoja.

V pričujočem članku smo se omejili na okoljske vidike trajnostnega razvoja. Zavedamo se, da so poleg okoljskih vidikov trajnostnega razvoja izjemno pomembni tudi socialni, o katerih podjetja tudi poročajo. Obe skupini vidikov sta v marsičem povezani in medsebojno odvisni, verodostojno trajnostno poročanje pa mora seveda zajeti tako ene kot druge vidike. Eden glavnih izzivov v prihodnje bo zagotovo tudi, kako spremeniti potrošniško kulturo v tem tržnem segmentu.

Verjetno bi k večji dinamiki in intenziteti ukrepov trajnostnega razvoja pripomogle aktivnosti, povezane s pridobitvijo certifikatov ISO 14001 ali EMAS, ki od podjetij zahtevajo vzpostavitev aktivne okoljske politike. Razen podjetja Adidas namreč proučevana podjetja na dostopnih poročilih in na spletnih straneh ne poročajo, da so si katerega od omenjenih certifikatov pridobila.

Pričakujemo lahko, da se bodo raziskave o trajnostnem poročanju v prihodnje še okrepile. Naša raziskava je prispevek k boljšemu razumevanju teh sodobnih trendov. Rezultati prinašajo nova spoznanja in vpogled v strategije trajnostnega poročanja podjetij v tekstilni panogi s poudarkom na proizvodnji in prodaji oblačil. Dodatno prinašajo v slovenski prostor informacije za bolj jasno razumevanje pomembnih trendov v panogi.

Pričujoča raziskava ima tudi določene omejitve. Treba je poudariti, da je bil naš vzorec raziskovanja relativno majhen, zato rezultatov ne moremo posploševati na celotno tekstilno panogo. Prav tako smo se v raziskavi omejili zgolj na velika in tuja podjetja s specifičnim poslovnim modelom s poudarkom na oblačilnih izdelkih. Čeprav so v dobavnih verigah tovrstnih izdelkov zajete vse faze tekstilne panoge (vključno s proizvodnjo), pa ima oblačilni sektor svoje specifike, zato rezultatov raziskave ni mogoče neposredno prenašati tudi na preostale sektorje tekstilne panoge.

Za celovitejšo sliko bi bilo treba proučiti tudi razvoj in implementacijo ekoinovacij v tekstilni panogi (produktnih, proizvodnih, organizacijskih) in poiskati korelacije s pristopi pri trajnostnem komuniciranju. Ker sta namen naše raziskave izključno pregled in analiza trajnostnih poročil, se z opisom, trendi in vrstami ekoinovacij v pričujočem prispevku ne ukvarjamo. Vsekakor pa iskanje korelacij med ekoinoviranjem in trajnostnim poročanjem prispeva dodatna pomembna spoznanja pri razumevanju odziva podjetij na zahteve trajnostnega razvoja. Zanimivo področje raziskav v tem kontekstu bi bilo tudi iskanje odgovorov, kako se na okoljsko problematiko odzivajo potrošniki, saj so eden ključnih elementov pri spreminjanju sedanjih tržnih praks.

Viri

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Influence of Ink Curing in UV LED Inkjet Printing on Colour Differences, Ink Bleeding and Abrasion Resistance of Prints on Textile

Vpliv sušenja tiskarske barve v UV LED kapljičnem tisku na barvne razlike, razlivanje tiskarske barve in odpornost proti drgnjenju potiskanih tkanin

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Abstract

Digital printing techniques are increasingly present in the field of textile printing. Particularly prominent is the inkjet printing technique using water-based inks, while UV LED inkjet printing also increasingly being in use. UV LED inkjet is primarily not intended for direct clothing printing; however, it can be used especially as a hybrid solution in the soft signage market. It is a great option for the printers that are not engaged only in textile printing, and want a more versatile print portfolio, extending it to non-clothing textile products, e.g. soft signage and non-wearable products. As these types of products often require colour reproduction of logos, accurate colour reproduction, good ink adhesion and sharpness are important just like in other printing technologies. In order to evaluate the impact of UV LED radiation amount on colour differences, ink bleeding and abrasion resistance, six different fabric samples (five woven and one nonwoven) were printed using a UV LED inkjet printer. Based on the results of colour difference, it was established that a reduction of UV radiation (by half the manufacturer's recommended amount) had no effect on this parameter. However, perceptible colour differences were observed with the use of different M measurement conditions defined by the international standard ISO 13655-2017. Reducing the amount of UV radiation had no effect on the adhesion and durability of the printed ink. Small differences detected in these two parameters were mainly a consequence of the properties of textile materials and not of decreased UV radiation.
Keywords: UV LED inkjet printing on textile, ink curing, ink bleeding, colour differences, abrasion resistance

Izvleček

Digitalne tiskarske tehnike so čedalje bolj prisotne na področju tekstilnega tiska. Pri tem ima vodilno vlogo predvsem kapljični tisk s tiskarskimi barvami na vodni osnovi, čedalje bolj pa je prisoten tudi UV LED kapljični tisk. Čeprav njegov prvotni namen ni tiskanje oblačil, je hibridna rešitev za tiskarje, ki poleg tekstilnih potiskujejo tudi druge vrste materialov. Med nabor tekstilnih izdelkov, ki se lahko tiskajo z uporabo tehnologije UV LED kapljičnega tiska, spadajo predvsem

neoblačilni izdelki in t. i. mehke oznake. Tudi pri teh so natančna barvna reprodukcija, obstojnost in kakovost odtisov ključnega pomena. Da bi ovrednotili vpliv sušenja tiskarske barve, ki je eden ključnih procesov v UV LED kapljičnem tisku, smo med raziskavo z različnima količinama UV-sevanja zamreževali oz. sušili tiskarsko barvo, odtisnjeno na šestih (petih tkanih in enem netkanem) tekstilnih vzorcih. Na podlagi rezultatov meritev barvnih razlik smo ugotovili, da zmanjšanje količine UV-sevanja (za polovico glede na tisto, ki jo priporoča izdelovalec tiskalnika) ni vplivalo na ta parameter. Sorazmerno velik vpliv na barvne razlike odtisov pa smo zaznali ob uporabi različnih M merilnih pogojev, ki jih definira mednarodni standard ISO 13655-2017. Zmanjšanje količine UV-sevanja ni vplivalo na adhezijo in obstojnost odtisnjene tiskarske barve. Majhne razlike, zaznane pri teh dveh parametrih, so bile predvsem posledica lastnosti tekstilnih materialov in ne posledica zmanjšanja količine UV-sevanja.

Ključne besede: UV LED kapljični tisk na tekstil, sušenje tiskarske barve, razlivanje tiskarske barve, barvne razlike, odpornost proti drgnjenju

1 Introduction

The presence of digitisation has been on an increase in virtually every industrial sector in recent years, which is also reflected in the field of textile printing. Digital printing techniques are becoming each year more prevalent in this segment, as evidenced by the Smithers Pira data, which predict more than 10% annual growth by 2023 [1]. Digital printing techniques are replacing analogue printing with the predominance of inkjet printing. Inkjet printing is based on spraying tiny droplets (with a volume of few picolitres) of liquid ink onto the printing substrate. The droplets are placed with great precision, enabling reproduction of high-quality images. The inkjet printing market share is growing mainly because it offers an economic alternative to other print techniques, having the advantage of full variability and low set-up costs. It allows economic printing of single copies on virtually any flat or flexible printing substrate (stone, polymers, glass, ceramics, composites, textiles etc.) [1, 2].

The dominant water-based technologies (with reactive, acid or pigmented inks) have the largest market share, while UV inkjet is a subtype of inkjet printing which is not intended for direct clothing printing but can be used especially as a hybrid solution in the textile soft signage market. It is a great option for printers that are not engaged only in textile printing and want their versatile print portfolio to extend to non-clothing textile products, e.g. soft signage and non-wearable products. As soft signage often includes colour reproduction of logos, an accurate colour reproduction is important just like in other printing technologies [3]. The main disadvantage of UV LED inkjet is the possibility of ink components migration and reaction with the human skin when used for wearable textile printing. Its greatest advantage, however, is that the printing ink dries as soon as it is applied onto the printing substrate and cured with a UV LED lamp. This enables printing with a greater amount of printing ink (better surface coverage and a more accurate colour reproduction with less influence of the printing material) and, above all, printing on (all) non-absorbent printing materials [2, 3]. Immediate drying is enabled by the photoinitiator, a component of printing ink that encourages the crosslinking of the printed layer of ink under the influence of UV radiation from lamps, which are a part of printers. Currently, the following photoinitiators are mainly in use: benzyl dimethyl ketal, 2-hydroxy-methyl-1 phenyl propane and hydroxycyclohexyl phenyl ketone. As a source of UV radiation, LED lamps are primarily used on modern printing devices. They use significantly less energy than conventional mercury lamps while producing less harmful ozone. Their advantage is also the narrower radiation range, with usually only one dominant peak located in the UV A region (320–395 nm) [2].

Two books by Ujiie [4] and Cie [5], along with an article by Malik, Kadian and Kumar [6] defined digital printing technologies and especially inkjet printing in detail, providing an overview of technical specifications of different parameters involved in the printing of textiles. When printed, UV inks are very low in viscosity and penetrate deeply into the fabric to adhere to the surface. The ink must be exposed directly to UV light to obtain a cure from a UV bulb. UV curable inks and their applications in industrial inkjet printing are described also in the book by Zapka [7] where the author mentions that the UV exposure time can influence image quality. An overview of influencing factors is presented in Figure 1.

Regarding published research, the UV inkjet printing on textiles (esp. UV LED inkjet) has not been widely

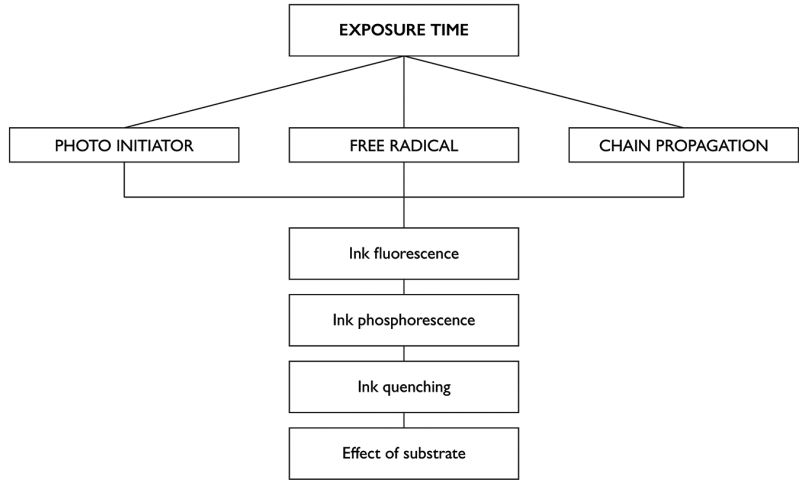

Figure 1: Factors in UV LED inkjet printing influenced by exposure time [7]

researched yet. The article by Hankock and Lin [8] covered the production of UV inkjet inks, while in the paper by Edison [9], the optimisation of UV curable inks was defined in terms of optimal jet output. Yi et al. [10] investigated the importance of monomers and comonomers in UV LED inks. Their work indicates that the monomer has not only a substantial influence on the dispersion and cure rate of UV LED ink but also a major effect on the film-forming properties of the ink. The mechanisms of attachment to textile fibres primarily include the interaction of chemical binding, mechanical interaction and fibre structure diffusion.

The influence of industrial fabrication parameters on the crosslinking density of UV resin was studied by Seipel et al. in 2018 [11]. A UV responsive smart textile was produced with inkjet printing and UV LED curing of a specifically designed photochromic ink on a PET fabric. The authors found out that increased ink deposition, or curing with higher intensity, i.e. higher lamp intensity and/or lower belt speed, increased the crosslinking density of ink. Hence, it formed a thicker or more distinct layer on the PET fabric surface. The effect of the deposited ink amount and curing settings on print durability is also described in this paper. A higher polymer crosslinking density is achieved as the print creates a strong insulation layer on the PET surface. The prints cured with the lowest curing intensity exhibited a lower polymer crosslinking density; however, they were slightly less durable and flexible. Mikuž et al. [12] compared the properties of inkjet printed, ultraviolet cured pigment prints with screen-printed, thermo-cured pigment prints. The colorimetric parameters of printed fabrics showed minimal and acceptable differences. A comparison of colour fastness properties proved that good colour fastness is achieved on pigment-printed fabrics produced with both printing techniques. The flat-screen-printed fabrics had better colour fastness to washing, perspiration and rubbing, while inkjet-printed fabrics showed better colour fastness to dry-cleaning and light. Tse et al. [13] studied the usability of image-based instruments for print quality evaluation. Regarding colour quality, the test results indicate that the fabric structure, yarn size and the hydrophilic/hydrophobic aspect of the fabric are the most important variables. Moreover, it was established that the colour gamut for larger size yarn is greater than for smaller size yarn and that there was an apparent downshift in the a*–b* plane for the knitted sample, indicating a colour shift between the two types of fabric structures. Bae, Hong and Lamar [14] found out that the texture of woven textiles caused a measurable effect on colour in inkjet printing, both using instrumental and perceptual measures. Colour reproduction is not only characterised by the interaction between light, dyes, pigments and textile structure, but also by the measurement conditions and geometry, and by the multi-layering of inks and process parameters. The multi-layering of inks and process parameters, e.g. washing fastness of printed inks, were studied by Kašiković et al. [15] in 2018. Two commercial spectrophotometers with different measuring geometries were used in a paper written by Milić et al. [16] to determine the measuring uncertainty of spectrophotometric measurements of print-

ed textile materials. Study findings suggest that, despite different measuring geometry, instruments had similar measurement repeatability behaviour (repeatability of readings from different parts of the same sample) in the case of used digitally printed polyester materials. The material preparation process (material was folded three times and placed on a black or white backing) had an important influence on measurement variability. In the recent study by Karlovits, Lavrič and Kavčič [17], four differently structured textile materials were printed with a UV LED inkjet printer. The spectrophotometric measurements of prints were conducted according to ISO 13655:2017 [18]. The obtained results revealed that the texture and aperture size had influence on colour differences, while the measurement mode differences were more prominent in the areas with higher than lower ink coverages, especially when using the polarisation filter for ink coverages over 150%.

Even though UV curing is one of the key processes in UV LED inkjet printing, its influence on print properties has not been widely researched in the literature. The aim of the research, therefore, was to describe the influence of the UV LED radiation amount on colour differences, ink bleeding and abrasion resistance of prints printed with a UV LED inkjet printer on six different fabric samples. The study also evaluated the influence of M mode measurement conditions on colour differences in the UV LED inkjet printing. The M mode measuring conditions are defined by the international standard ISO 13655:2017 [18] and are widely used especially for paper and cardboard printing applications. They are a response to the increasing presence of optical brighteners in papers and cardboards, which creates challenges for successful colour management and accurate colour reproduction. Optical brighteners are chemical substances added to different materials (e.g. paper, board, fabrics) to enhance their brightness. They absorb invisible ultraviolet radiation at wavelengths below 400 nm and emit it in the blue end of the visible spectrum at approximately 400 to 450 nm through an electrophysiological alteration (fluorescence process). This process is activated only when the M1 measuring condition is used for spectrophotometric measurements. By choosing this measurement condition, the measurement is performed using the D50 illumination condition with the UV component of light included. At the M2 measuring condition, this part of the light is excluded (UV cut), while the measurement condition M0 is based on the measurements with the Standard Illuminant A. As by scope, ISO 13655:2017 is not applicable just for paper and board types of substrates, and there is no other specific standard regarding the measurement conditions for digitally printed textiles. It is more common that the printer will use a spectrophotometer, which covers more types of printing materials, and these differences are important for evaluation.

2 Materials and methods

2.1 Materials

The influence of UV curing in the UV LED inkjet printing on colour differences, print sharpness and abrasion resistance was evaluated on six different fabric samples (5 woven and 1 nonwoven). Their properties are presented in Table 1.

Printing forme preparation

A digital printing forme (cf. Figure 2) was designed using the computer program Adobe Illustrator CC (Adobe, USA) and saved as PDF without the colour

Table 1: Sample properties

Sample	Weave type (ISO 3572:1976)	Thread count warp/weft (ISO 7211-2:1984)	Thickness (mm) (ISO 5084:1996)	Composition (ISO/TR 11827:2012)	Optical brighteners (ISO 3664:2009)	Mass per unit area (g/m²) (ISO 3801:1977)
V1	plain weave	25/21	0.354	100% CO	YES	134
V2	plain weave	51/30	0.228	100% CO	NO	121
V3	plain weave	35/21	0.316	50% PES/50% CO	NO	171
V4	plain weave	24/21	0.460	100% PES	YES	204
V5	crepe weave	30/21	0.573	100% PES	NO	199
V6	fleece	/	1.170	100% PES (nonwoven)	NO	151

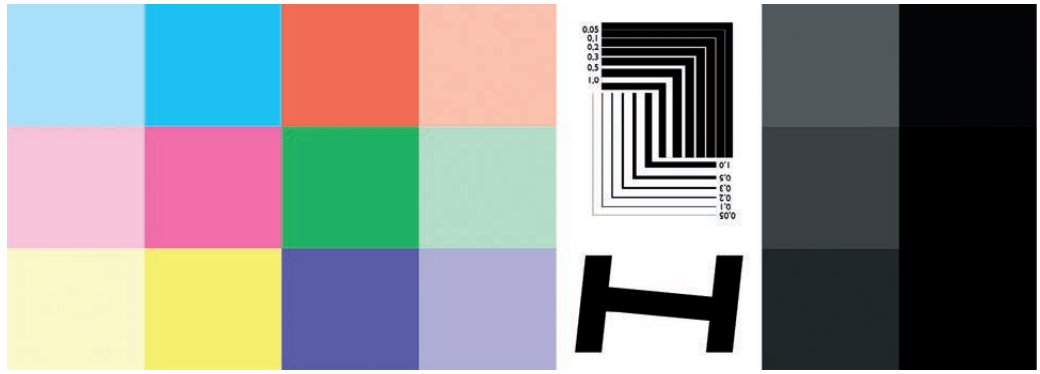

Figure 2: Digital printing forme

profile attached. It consisted of 18 colour patches with the area of 4 cm² (CMY patches with 50% and 100% tone value; RGB patches with 50% and 100% tone value, where e.g. 50% R is defined as 50% M + 50% Y and 100% R is defined as 100% M + 100% Y etc.) The total ink coverage scale consists of patches in which the tone values gradually increase (from 50% C + 50% M + 50% Y + 50% + 100% B to 100% C + 100% M + 100% Y + 100% B), lines and a control element for print sharpness evaluation. The file was then processed using a SAi PhotoPRINT DX Plus (SAi, USA) raster image processor. Linearization without colour corrections was performed.

Printing process

The samples were printed with an Apex UV 1610 UV LED flatbed inkjet printer (Apex, China) equipped with Toshiba CE4 on-demand piezo electric inkjet print heads and two UV LED lamps (one on the left and one on the right side of print heads). Sakata soft LED UV inks (Sakata, Japan) were used. They were formulated to cure when exposed to UV light with the wavelength of 395 nm. The printing parameters were set to 8 passes, with the printing speed of 0.84 m/s (one-directional printing – from right to left). The print head height was set 0.8 mm above the material top and the jetting frequency to 10.28 kHz. The printing ink drop size was 6 picolitres. The ink was cured using one or both UV lamps. Five prints were made on each fabric sample.

Spectrophotometric measurements and colour differences calculation

Spectrophotometric data were obtained using a spectrophotometer X-Rite i1 Pro 2 Basic (X-Rite, USA) and BabelColor PatchTool (BabelColor, Canada) software. The measurements were performed with the M0, M1 and M2 measurements modes (cf. Table 2) on standardised white backing. The measuring conditions were set to 45°/0° ring illumination optics, D50 standard illuminant and 2° standard observer. Colour differences (ΔE_{00}) were calculated using BabelColor PatchTool software in accordance with ISO 13655:2017 [18]. Five measurements were made on each colour patch.

Ink bleeding evaluation

The ink bleeding evaluation was done following the method described by Hladnik and Muck in 2011 [19]. It is based on the measurements of the area (mm²) and perimeter (mm) of a selected printed element that are compared with the measurements of its undistorted digital form from the printing form. The measurements were conducted using an ImageJ 1.48v (ImageJ, USA) computer program on TIFF images with the resolutions of 600 ppi obtained with CanoScan 5600F (Canon, Japan) without any colour distortions and corrections.

Crockfastness evaluation

Crockfastness was measured according to ISO 105-X12:1993 on a CM-5 Crockmeter (AATCC Atlas,

Table 2: Description of measuring conditions

Measuring condition	Light source	Filter
M0	undefined/tungsten	none
M1	D50 + controlled UV	none
M2	tungsten	UV cut

USA). Ten measurements were performed for dry and wet crockfastness tests.

Colour fastness to washing

Colour fastness to washing at 40 °C was tested in accordance with ISO 105-C06:2010.

FTIR ATR analysis

The FTIR ATR printing ink analysis was performed using a Perkin Elmer Spectrum Two FTIR spectrometer (Perkin Elmer, USA). For the purpose of the analysis, printing ink was printed on an inert glass surface with one and two UV lights used for curing, and then peeled from it and analysed. In this way, a potential impact of the textile on the analysis was nullified.

3 Results and discussion

Table 3 shows the average values of colour differences among the prints printed using one or two UV lamps on each textile sample. Colour differences were calculated based on the spectrophotometric values of all colour patches on the printing form. All patches were measured under the M0, M1 and M2 measuring conditions.

Based on the results shown in Table 3, it can be concluded that between two different amounts of UV radiation, there was a minimal effect on the colour reproduction of textile samples. The average calculated colour difference was 0.55 ΔE_{00}. Such a colour difference is almost unnoticeable to the human eye and is most likely a consequence of short-term repeatability of the measuring instrument and the printer. Despite the 50% reduction in UV radiation from the radiation recommended by the printer manufacturer, it still polymerised the ink and thus prevented further penetration, which could lead to greater colour variations. However, this was not fully confirmed by the FTIR analysis, the result of which is shown in Figure 3.

As it can be seen from Figure 3, the sample of printing ink that was less crosslinked (red curve) achieved slightly higher absorbance values across the entire spectrum (from 450 to 4000 cm^{-1}) than the sample that was crosslinked to a greater extent. The vertical shift of curves can be attributed to the difference in the amount of twisting and stretching vibrations

Table 3: Average colour differences (ΔE_{00}) among prints printed using one and two UV lamps

Sample	Measuring condition		
	M0	M1	M2
V1	0.51	0.51	0.53
V2	0.30	0.29	0.30
V3	0.69	0.69	0.69
V4	0.56	0.55	0.58
V5	0.52	0.52	0.52

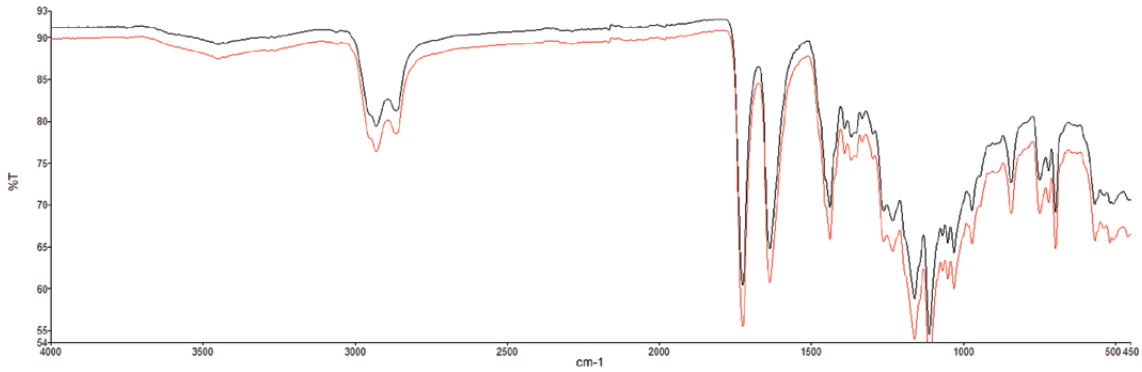

Figure 3: FTIR spectra of black process printing ink cured with one (red curve) and/or two UV LED lamps (black curve)

between molecules and atoms, which was more noticeable at the less crosslinked sample of printing ink. The difference is clearly noticeable also at approx. 807 cm^{-1}, which indicates a greater presence of C=C bonds in the less crosslinked sample. The amount of unreacted C=C double bonds between acrylic molecules is a direct indicator of UV ink polymerisation (several unreacted C=C double bonds indicate a lower degree of ink polymerisation).

A much greater influence on colour differences, especially for samples V1 and V4, which contained optical brighteners, can be observed due to the selection of measuring conditions. The results shown in Table 4 were obtained by measuring the prints dried using two UV LED lamps, the only variable factor being the choice of the measurement condition.

Optical brighteners present in textile samples (V1 and V4) affected the colour reproduction significantly (cf. Table 4). This is the main reason for relatively big colour differences (on average between V1 and V4; M0 : M1 1.10, M0 : M2 2.98 and M1 : M2 4.07 ΔE_{00}). The majority of colour differences (more than 60%) were detected in the least covered fields (50% CMY). Mainly due to the properties of tested materials (structure and weaving), spectrophotometric measurements were also influenced by optical brighteners in more covered fields or patches (the influence of optical brighteners present not only on the surface of the fibres but also on their circumference). Such colour differences are perceptible through close observation (M0 : M1) and at a glance in the case of comparing M2 measuring conditions with M1 and M0. The colour differences of samples without optical brighteners were negligible. Negligible were also the differences in the area and perimeter of selected printed elements which were measured for printing sharpness determination and are presented in Table 5.

Table 4: Colour differences (ΔE_{00}) caused by selection of measuring condition

Sample	Measuring condition		
	M0 : M1	M0 : M2	M1 : M2
V1	1.13	3.03	4.15
V2	0.01	0.03	0.04
V3	0.02	0.05	0.06
V4	1.07	2.92	3.99
V5	0.02	0.05	0.07

Table 5: Areas and perimeters of selected printed element

Sample	Curing – number of active lamps while printing	Area (mm^2)	Perimeter (mm)
Ideal, digital element	/	190	115
V1	1	187.8	114.9
	2	188.2	114.3
V2	1	191.0	115.2
	2	190.6	114.2
V3	1	193.7	117.4
	2	194.7	118.5
V4	1	189.8	113.9
	2	189.2	114.9
V5	1	187.6	115.7
	2	189.4	117.5
V6	1	190.6	121.4
	2	190.4	122.4

The differences in areas and perimeters caused by lower UV radiation are minimal and can be attributed to the deviation of the method. Despite the one lamp being turned off, a sufficiently strong crosslinking occurred quickly enough for the printing ink not to spill or bleed. The differences among the samples, however, can be attributed to their different structure. Table 6 represents the colour fastness properties of samples to washing at 40 °C. Reduced ink curing did not influence this parameter on woven samples, while it improved colour fastness to washing of the nonwoven sample V6. The absence of empty spaces between the threads in this sample retained a greater amount of printing ink on the fabric surface. The ink layer was slightly more flexible with reduced curing. This affected the result of colour fastness to washing; however, it did not affect the results of the Crock test shown in Table 6.

The results of the crock test (cf. Table 7) were not affected by the reduction in curing. Small differences can be attributed to the standard deviation of the method, which is based on the visual assessment. Among the samples, the worst result was achieved with the nonwoven sample (V6). The printing ink layer, which remained on the surface of the sample, was more exposed when rubbed than in other samples.

4 Conclusion

Reducing the amount of UV radiation used for printing inks curing (by half the manufacturer's recommended amount) had no significant effect on the colour differences and print sharpness of printed textiles. A selection of different measuring conditions, however, caused perceptible colour differences in two samples which had optical brighteners, the other four had no significant changes. The difference between the applied two levels of radiation did not influence the ink properties, and thus the differences due to additional ink fluorescence and other optical effects. The FTIR analysis showed a difference in the degree of polymerisation of the printing ink, which was cured with one or two lamps, however, from the applicative point of view, the difference proved to be practically irrelevant. It is very important that the presence of optical brighteners is taken into account and that modern and calibrated measuring instruments are used for colour measurements. The

Table 6: Colour fastness to washing at 40 °C

Sample	Colour fastness to washing 2 UV lamps	Colour fastness to washing 1 UV lamp
V1	4/5	4/5
V2	4	4/5
V3	4/5	4/5
V4	3	3
V5	5	5
V6	4	5

Table 7: Crock test evaluation

Sample	Grade			
	Dry 1 UV lamp	Dry 2 UV lamps	Wet 1 UV lamp	Wet 2 UV lamps
V1	3	3	3	2/3
V2	2/3	2/3	3	3
V3	2/3	2/3	2	2
V4	2/3	2/3	1/2	1/2
V5	2	2	1/2	1/2
V6	2	2	1	1

adhesion and fastness tests resulted in small, insignificant differences, indicating that the drying can be done equally efficiently with just one lamp on textile materials. The differences in the crockmeter were mainly due to the material properties and not a result of drying exposure variation.

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Performance Evaluation of PLA Based Biocomposites Reinforced with Photografted PALF

Ocena učinkovitosti biokompozitov na osnovi polimlečne kisline, ojačenih s fotoinducirano cepljenimi ananasovimi listnimi vlakni

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Abstract

In this study, biocomposites were fabricated through a compression moulding technique that used untreated and grafted pineapple leaf fibre separately with polylactic acid (PLA) as a matrix. For grafting, pineapple leaf fibre (PALF) was chemically modified using two different monomers, i.e. 2-hydroxyethyl methacrylate (HEMA) and methyl methacrylate (MMA) solutions, in the presence of methanol (MeOH) and photoinitiator (Darocur-1664) under ultraviolet (UV) radiation with the aim of improving thermo-mechanical characteristics. Based on grafting efficiency and mechanical attributes, the intensity of UV radiation and monomer concentration were maximized. A series of solutions, created by varying the concentrations (10–60%) of monomers in MeOH along with 2% photoinitiator, were prepared. Experimental results revealed that composites made of PALF grafted with 30% HEMA at the 15th pass and 40% MMA at the 20th pass of UV radiation achieved the optimum mechanical properties compared with an untreated PALF/PLA composite. The optimized solutions were further enhanced by adding various concentrations (0.5–1.5%) of urea, with the best mechanical features achieved using a 1% concentration of urea. The chemical bonds formed due to photografting were viewed using Fourier transform infrared spectroscopy (FTIR). Degradation behaviour under heat was determined through thermogravimetric analysis, which found that photografted PALF/PLA showed significantly better thermal stability than the untreated composite sample. A water uptake test showed that grafting reduced the water retention capacity of the treated composite significantly. Crystallization characteristics were inspected using a differential scanning calorimeter, which showed that grafted PALF had a substantial effect on the degree of crystallization of PLA. In addition, scanning electron microscopy was used to monitor the interfacial bond, and revealed that interfacial adhesion was enhanced by the incorporation of photografted PALF into the matrix.

Keywords: photografting, PALF, thermo-mechanical properties, PLA, UV radiation

Izvleček

V tej raziskavi so bili iz neobdelanih oziroma površinsko aktiviranih listnih ananasovih vlaken in polimlečne kisline (PLA) kot matrice po postopku oblikovanja z vlečenjem izdelani biokompoziti. Za izboljšanje termomehanskih lastnosti so bila ananasova listna vlakna (PALF) kemično modificirana s pomočjo dveh različnih monomerov, in sicer raztopine 2-hidroksietil metakrilata (HEMA) in raztopine metil metakrilata (MMA) v prisotnosti metanola (MeOH) in fotoiniciatorja (Darocur-1664) ter uporabe ultravijoličnega sevanja (UV). Intenziteta sevanja UV-žarkov in koncentracija monomera sta bila optimizirana glede na učinkovitost cepljenja in mehanske lastnosti kompozitov. Pripravljena je bila serija 10–60-odstotnih raztopin monomera v metanolu z dodatkom 2-odstotnega fotoiniciatorja. Eksperimentalni rezultati so pokazali, da so optimalne mehanske lastnosti dosegli kompoziti, ojačeni s predhodno cepljenimi vlakni PALF s 30-odstotnimi HEMA in 15 cikli osvetljevanja z UV-žarki, medtem ko so kompoziti iz predhodno cepljenih vlaken s 40-odstotno raztopino MMA dosegli optimalne lastnosti po 20 ciklih osvetljevanja z UV-žarki. Optimiziranim raztopinam je bila dodana sečnina v 0,5–1,5-odstotnih koncentracijah, pri čemer so bile najboljše mehanske lastnosti kompozitov dosežene z uporabo enoodstotne koncentracije sečnine. Kemične vezi, ki so nastale zaradi cepljenja vlaken, so bile dokazane s pomočjo infrardeče spektroskopije s Fourierjevo transformacijo (FTIR). Termogravimetrična analiza je pokazala, da je kompozit PALF/PLA s cepljenimi vlakni imel bistveno boljšo toplotno stabilnost kot kompozit PALF/PLA z neobdelanimi vlakni. Prav tako je cepljenje vlaken znatno zmanjšalo sposobnost zadrževanja vode v kompozitu. Z diferencialno termično kalorimetrijo je bilo ugotovljeno znatno zvišanje stopnje kristaliničnosti PLA v kompozitu PALF/PLA, ki je vseboval cepljena vlakna. Poleg tega je bila za spremljanje medfazne adhezije v kompozitu uporabljena tudi rastrska elektronska mikroskopija, ki je pokazala povečanje adhezije z vključitvijo fotoinduciranih cepljenih vlaken PALF v matrico.
Ključne besede: fotoinducirano cepljenje, PALF, termomehanske lastnosti, PLA, UV-sevanje

1 Introduction

Interest in plant extracted lignocellulosic fibres as a reinforcing filler in composites has risen significantly during the last few decades for environmental reasons. However, research carried out in this area has determined that the integration of lignocellulosic fibres enhances the qualities of plastics. Plant-based fibres showed some notable benefits when compared to artificial fibres. For instance, they have comparable physico-mechanical properties, are inexpensive, cause no skin irritation, consume a small amount of energy during production and supply more O_2 to the environment, and emit lower amounts of CO_2 and toxic fumes during heat treatment, while the most prominent property is that they are renewable and decomposable. Due to their eco-friendly nature, these natural fibres are used in engineering applications in numerous sectors, such as the textile, automotive, aeronautic and construction industries, and in biomedical sectors [1–9], and thus encourage scientists to search for more and new classes of green and sustainable materials. In this regard, polylactic acid (PLA) is preferred for accelerating biodegradation in composites as a matrix because it is a biopolymer sourced from renewable products, it facilitates processing and has high thermo-mechanical properties compared to other thermoplastics, in combination with reduced manufacturing costs. PLA-based composites reinforced with plant-sourced bast fibres have already been studied, although much variation has been found in their properties. To establish suitable applications of composites made with PLA, further enhancement is required through reinforcement, where natural fibres are preferred for that purpose [10, 11].

This study focuses on lingo-cellulosic pineapple fibre, which can serve as a favourable reinforcement, as it is abundantly available in tropical countries. Pineapple fibre is collected from the leaves of the pineapple plant, which are often discarded after the collecting of fruits. Currently, pineapple leaf fibre (PALF) holds no commercial value except for the nutritional purpose of its fruit, and is considered agro-waste. It is thus necessary to develop the standard of PALF by enhancing the physico-chemical and thermo-mechanical properties that might exaggerate the demand for consumption of the fibre. A study of PALF showed that it possesses outstanding thermal and mechanical characteristics that are equivalent to common lignocellulose fibres, such as jute, ramie and hemp, which are already established and extensively used as reinforcements in composite materials [12]. The fibre contains 67–82% cellulose, 18.8% hemicellulose, 4–15% lignin, 1–3% pectin, 4% waxing material, and a small amount of ash (3%). The density and diameter range is 1.07–1.52 g/cm3 and 20–80 μm respectively, together with a tensile strength (TS) of 413–1627

MPa, a Young's modulus (YM) of 34.5–82.51 GPa and elongation at break (Eb) of 1.6–3% [13, 14]. Thus, cellulose is the main component of PALF constituting anhydro-glucose units (1, 4–β conformation). These units comprise –OH groups that are mainly responsible for the higher moisture take up of PALF as the main drawback relative to other plant-extracted natural fibres. Consequently, chemical treatment is crucial for enhancing the characteristic properties of the fibre, so that physical, mechanical and thermal properties, as well as sustainability, will be superior while at the same time preserving its environment-friendly property [15, 16].

For overcoming the problems associated with natural fibres, many researchers have attempted to develop existing properties using different chemical treatments, such as change of functionality, graft copolymerization and acetylation, with the aim of improving its quality and genetically enhancing end products for diversified applications in numerous fields [17]. One of the most successful methods for developing the physico-mechanical behaviour of natural fibres is grafting with vinyl monomers under a radiation-induced method [18]. Radiation processing technology is a convenient way for grafting and modifications due to the introduction of stronger cross-linking through the rapid free radical propagation reaction of the multifunctional vinyl monomer, along with the reduction of the hydrophilic nature of cellulose fibre [19]. Over the decades, the impact of radiation on polymeric substrates, predominantly UV and γ (gamma), has been examined comprehensively. These radiations create ionization through the production of electrons, ions and free radicals [20]. Photo cross-linkable polymers contain functional groups that can directly form a cross-linked polymer through light-induced reactions. Photoinitiators have numerous applications in photo-induced polymerization. The benefits of photocuring treatment in polymers include improved monomer stability, a significant reduction in reaction time, low energy consumption, etc. [21, 22]. Numerous studies have already been performed on monomer-based grafting with cellulosic fibres [23–25] to enhance potentialities under certain environmental conditions, particularly on jute, which has already gained considerable interest from many researchers. However, fewer reports can be found on radiation and photocured grafting. Nevertheless, sounder study, noticeably skilful efforts and cautious experimental techniques are required to achieve the full commercial benefits in this area.

The present study involves the modification of PALF to optimize the fibre's attributes with the aim of broadening its future use in industrial applications. For this purpose, PALFs were treated with two types of acrylic monomers under various intensities of UV radiation, and the resulting treated fibres were further set to produce PALF/PLA composites. A brief examination was carried out on thermo-mechanical properties of the treated composites and further proved that the composites can be used for diversified applications with better serviceability.

2 Experimental

2.1 Materials

PLA pellets and PALF were supplied from DS fibres, Belgium and Madhupur, Bangladesh respectively. The molecular weight and density of PLA was 209 kDa and 1.273 g/cm^3 respectively. Two types of monomer 2-hydroxyethyl methacrylate (HEMA), methyl methacrylate (MMA) and the swelling agent methanol (MeOH) were acquired from the German Company E. Merck. Darocur–1664, whose function was as photoinitiator, was purchased from Ciba-Geigy. The structures of the used monomers are shown in Figure 1. Urea, which was used as an additive and acetone (CH_3COCH_3), was purchased from British Drug House, UK.

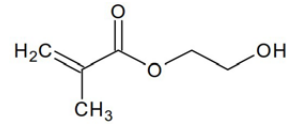
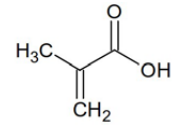
Monomer	Chemical structure
2-hydroxyethyl methacrylate (HEMA)	
Methyl methacrylate (MMA)	

Figure 1: Chemical structure of the monomers used in this study

2.2 Evaluation of fibre properties

Fibre density was measured using the simplest method established by Archimedes where no equipment is required. At first, an open air-dried sample is weighed and weighed again after being dipped in a degassed fluid whose density was predetermined. The change in mass is referred to as buoyant force, which is then converted to specimen volume by dividing the fluid density. Finally, sample density was calculated by dividing the open air-dried sample weight by volume. This type of calculation is covered by the ASTM D3800 standard [26].

The ISO 1973:1996 standard was used to determine the linear density of the fibres. Ten fibre tufts with a mass of several milligrams were taken from the sample and the fibres of each tuft were brought into parallel arrangement according to the relevant method. The middle part of each combed tuft was 50 mm in length. Five fibres were taken from each of ten bundles in turn, so as to form a bundle of 50 fibres. Ten such bundles were made. These bundles were weighed individually using a scale, to an accuracy of 0.1 mg. The following equation was used to determine the mean linear density of fibre in each bundle.

$$L = \frac{m}{n \times l} \times 10^4 \quad (1)$$

where, L represents the mean linear density of the fibre in each bundle (dtex), m represents the mass of fibre bundle (mg), n represents the number of fibres in the bundle and l represents the length of the individual fibres in the bundle (mm).

Firstly, single fibres were separated manually from the bundle of pineapple fibres. The mechanical properties of individual fibres were determined according to the ASTM D3822 standard. The tests were done using universal Titan SN1410 series (James Heal) testing equipment. During testing, a load cell of 20 N and a gauge length of 10 mm were maintained. The test speed was 2 mm/minute. Five specimens were tested for each type of test. The data presented in Table 1 are the average values of the five tests for each case. Some properties of the used fibre are presented in Table 1.

2.3 Grafting of PALF with monomers

PALFs were first cut into the desired length (20–25 mm). To eliminate foreign components and dirt, the fibres were washed with acetone followed by drying at 105 °C in an oven and then weighed. The monomers (HEMA, and MMA) were used independently in various concentrations (10–60%) in the formulation.

Table 1: Physico-mechanical features of used PALF in the study

Properties	PALF fibre
Density (g/cm^3)	1.523 ± 0.35
Linear density (dtex)	6.8 ± 0.78
Tensile strength (MPa)	182.7 ± 7.6
Young's modulus (GPa)	6.347 ± 0.87
Elongation at break (%)	2.75 ± 0.45

A 2% photoinitiator (Darocur-1664) and methanol (MeOH) were incorporated into the final composition. For swelling the cellulose backbone with the aim of improving impregnation, the monomers were appropriately mixed individually with methanol and the formulated solutions were stirred continuously for 1 hour to eliminate bubble formation. The resulting compositions are shown in Table 2. The dried virgin PALFs were immersed for 10 minutes in these resulting solutions. The soaked fibres were then subjected to irradiation with UV light. A UV radiation source (Minicure-200, IST Technik, Germany) was used for the irradiation of the fibres at the wavelengths of 254–313 nm, together with 50A current operated at 2kW power. A conveyor belt (length of 1 m) present in the UV irradiator rotates at a speed of 4 m/minutes around a mercury lamp. One movement towards the light is considered one pass. Specimens were kept on the conveyor and allowed to pass continuously through the UV source, and the number of passes was recorded. Before testing, the irradiated samples were kept in a relaxed state for 24 hours. Grafting percentage was calculated from the weight gain basis principle:

$$\text{Grafting (\%)} = \frac{A - B}{B} \times 100 \quad (2)$$

where, A and B represent weight of the sample after and before treatment respectively.

2.4 Composite manufacturing process

2.4.1 Fabrication of PLA sheets

The PLA and PALF were dried properly at 80 °C for 10 hours in vacuum conditions to remove moisture and avoid the creation of voids during the manufacturing process. First, PLA films of 1 mm thickness were prepared from pre-weighed pellets using a compression moulding machine (Carver Inc. model 3856, USA) at a temperature of 190 °C for 10 minutes, applying

Table 2: Composition of different formulations (w/w %)

Materials	H_1	H_2	H_3	H_4	H_5	H_6
HEMA	10	20	30	40	50	60
MeOH	88	78	68	58	48	38
Darocur-1664	2	2	2	2	2	2
Materials	M_1	M_2	M_3	M_4	M_5	M_6
MMA	10	20	30	40	50	60
MeOH	88	78	68	58	48	38
Darocur-1664	2	2	2	2	2	2

a pressure of 50 bar. Cooling was done for 5–7 minutes at the same pressure using a different moulding machine. The PLA films were cut to the required size (18 cm × 18 cm) and weighed.

2.4.2 Fabrication of composite laminates

Prior to fabricating PALF/PLA composites, PALF fibre was cut to a length of 20–25 mm. Composites having 3 mm thickness were prepared by sandwiching three layers of PALF between four pre-weighed PLA sheets. The dimension of the mould was 18 cm × 18 cm × 3 mm for the preparation of composites. The sandwiched PLA sheets were then placed between two steel moulds with randomly oriented PALF and heated at 190 °C at a pressure of 50 bar for 10 minutes in a Carver heat press (shown in Figure 2). They were then allowed to cool by passing water from an inlet pipe over both upper and lower plates for 10 minutes. The composite sheet was then removed from the mould plate to undergo natural cooling for 30 minutes at room temperature. Composites were manufactured using untreated and monomer treated fibres separately by maintaining the formulations shown in Table 3. The composite samples of the required dimensions were then cut carefully with a grinding machine from a large composite sheet for the purpose of determining different physical and mechanical properties.

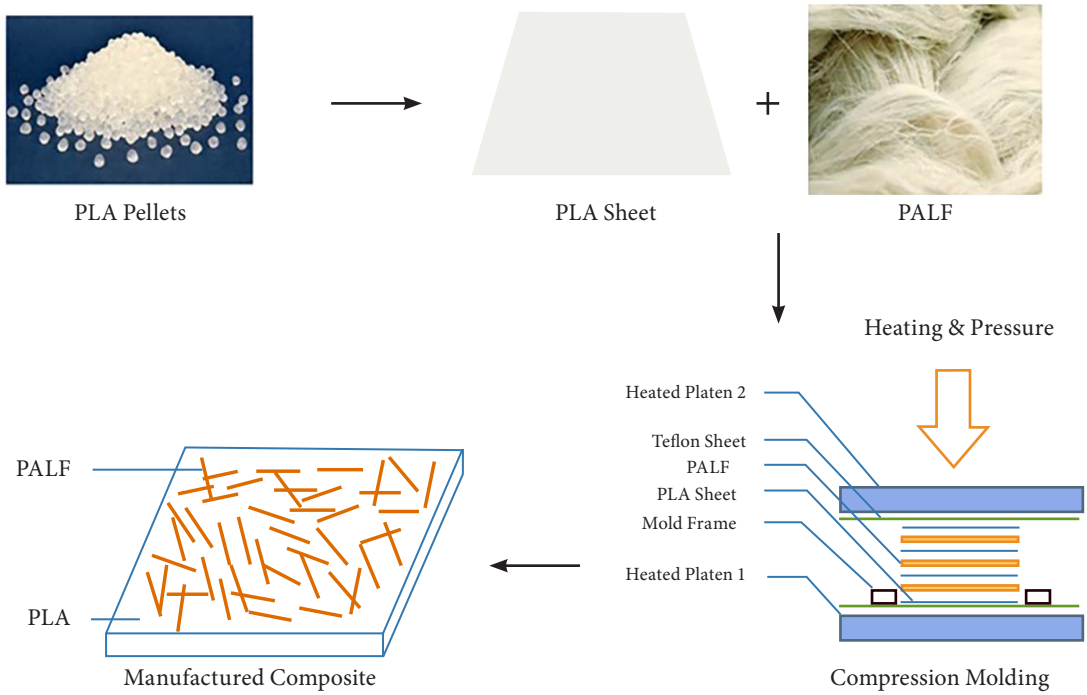


Figure 2: Fabrication model of PALF/PLA composites by compression moulding

Table 3: Composite formulations

Composite termed as	Type of treatment on fibre	Composition (PALF: PLA)
UC	Untreated	40:60
HC	30% HEMA treated (H3 sample)	40:60
MC	40% MMA treated (M4 sample)	40:60

2.5 Mechanical testing of composites

The mechanical properties of composites were determined in accordance with the ISO 527-4:1997 standard using a universal testing machine (Instron 5569). The load was 10 kN with an extension rate of 2 mm/minutes using a 25 mm gauge length. The flexural properties were measured in accordance with the ISO 14125:1998 method with a different Instron (type 4411) machine using a load cell of 5 kN and a speed of 2 mm/minute. The Izod test method (BS EN ISO 180: 2000+A2:2013) was used to determine the impact property, followed by the use of an Avery pendulum impact tester with a specimen size of 80 mm × 15 mm × 3 mm. For tensile and bending tests, a sample size of 60 mm × 25 mm × 3 mm was maintained. To determine the final result, the mean value of 10 specimens was calculated at an accuracy of ± 0.5%. All of the experiments were performed under standard atmospheric conditions (25 °C ± 2 °C and 65% ± 2% RH). Table 4 shows the properties of PLA and virgin PALF/PLA composite.

Table 4: Mechanical behaviours of pure PLA and untreated PALF/PLA (UC) composite

Properties	Neat PLA	PALF/PLA
PALF fibre content (wt.%)	0.0	40
Tensile strength (MPa)	62.0 ± 2.5	111.7 ± 4.5
Young's modulus (GPa)	5.8 ± 0.5	10.8 ± 1.1
Flexural strength (MPa)	87.5 ± 3.5	175.0 ± 6.0
Flexural modulus (GPa)	6.3 ± 0.4	13.6 ± 0.7
Impact strength (kJ/m2)	6.9 ± 0.7	15.3 ± 1.5

2.6 Thermogravimetric analysis (TGA)

To investigate the thermal stability and decomposition configuration of PALF/PLA composites, thermogravimetry (TG) and derivative thermogravimetry (DTG) were carried out using a TA Instrument Q500 under nitrogen atmospheric conditions. For testing, small pieces of samples were prepared (10–20 mg wt.) and positioned into crucibles. The experiment was performed at temperature ranging from 0–600 °C with 5 °C/minute scan rate, and the corresponding weight loss (%) was recorded.

2.7 Differential scanning calorimetry (DSC)

Thermal behaviour and melting features of PLA and PALF/PLA composites were examined using a differential scanning calorimeter (model TA instrument DSC Q100) under nitrogen atmospheric conditions. The specimens were scanned from –20 to 220 °C at a heating rate of 10 °C/minute. The cooling rate was same as the heating rate. The following calculation was used to determine the degree of crystallinity:

$$X_c(\%) = 100 \times \frac{\Delta H_m}{\Delta H_f} \quad (3)$$

where ΔH_m represents the melting enthalpy of pure PLA and PALF/PLA composites and ΔH_f represents the melting enthalpy of 100% crystalline PLA (93.7 J/g).

2.8 Water retention test

To study the swelling behaviour of composites, a water retention test was performed to simulate the soaking phenomena of fibres. To determine the water retention capacity, a 5 g sample was immersed in a beaker holding 200 ml of deionized water. The samples were withdrawn periodically, wiped carefully and weighed. The water retention (%) was determined by means of the following weight gain formula:

$$W_z(\%) = [(W_x - W_y/W_y)] \times 100 \quad (3)$$

where W_z represents the water absorption quantity and W_x, and W_y represent the mass of the specimens before and after immersion.

2.9 Morphological studies

To observe the interfacial bonding of composites, fractured surface images were viewed using a scanning electron microscope (Philips XL30) with an acceleration of 5.0 kV.

3 Results and discussion

In this work, two types of acrylic monomers were selected to achieve enhanced features of PALFs by means of a radiation-induced graft copolymerization technique. A brief study was made of the impact of UV radiation and monomer concentration (%) on grafting and mechanical properties, the effect of additives on mechanical properties, and the water absorption of grafted PALF/PLA composites. The variation in thermal properties and morphological changes due to grafting and radiation were also studied.

3.1 Grafting (G_f)

PALFs were dipped for 10 minutes in solutions produced according to a predetermined formula at various concentrations (10–60%) of MMA and HEMA, followed by UV irradiation at different intensities (5–30 UV passes). The results are depicted in Figures 3 and 4, where grafting is shown against UV intensities with regard to monomer concentrations. The grafting percentage indicates the amount of cross-linkage produced between monomer and fibre. The grafting values were comparatively low at minor concentrations of monomer, and increased to a certain level of concentration before decreasing. The grafting (%) upsurge with the increase in UV dose reaches an extreme level at a definite intensity of UV and then declines as UV intensity is increased. However, the optimum grafting in most of the experiments was seen at the 15th UV pass for HEMA and the 20th pass for MMA treated fibre. After 20 passes, the amount of grafting decreases, possibly due to the degradation of the polymer under radiation. Nevertheless, the maximum grafting value (17.8%) was observed for 30% HEMA cured with 15 passes of UV radiation (H_3 sample). The optimum grafting occurs at the 15th pass (G_f = 15.7%) of UV radiation for MMA with a 40% concentration (M_4 sample), after which the grafting value falls, as seen in Figures 3 and 4 respectively. Hereafter, all tests were performed under these optimized conditions. Among the three monomers, HEMA treated fibres showed more grafting (%) than the MMA monomer, possibly due to its bulkier functional group with a long polymer chain. At a lower level of monomer concentration, free radicals form very quickly through propagation reactions. The photoinitiator strongly supports acrylic monomers to accelerate these reactions. Therefore, branched structures are produced by using double bonds during graft copolymerization. With an increase in monomer concentration, the residual unsaturation amount also rises, resulting in the quicker formation of 3-D network structures, which restricts mobility [27, 28]. After the achievement of the highest G_f (%), the amount of grafting was reduced with an increase in monomer concentration, which experimental data indicated might be the result of two main factors. At the upper level of concentration, the radical-radical formation reaction can be the result of recombination methods and additional homopolymers may instead generate the creation of monomer, together with cellulose. An additional factor may be the inadequate soaking of MeOH with the backbone

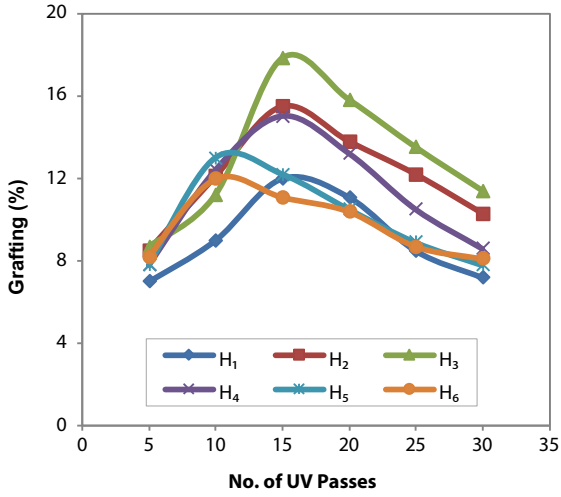


Figure 3: Grafting (%) of HEMA treated PALF against UV intensities as a function of various monomer concentrations

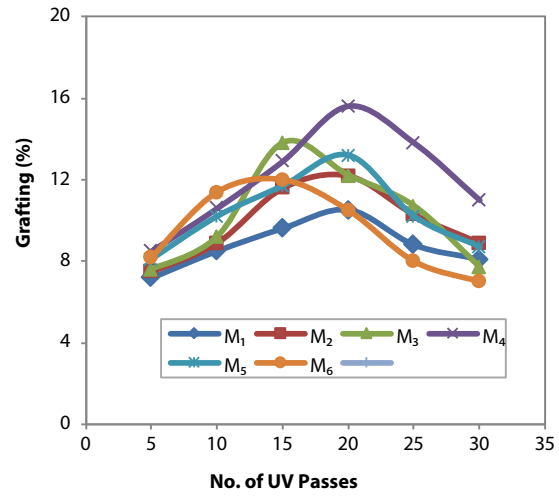


Figure 4: Grafting (%) of MMA treated PALF against UV intensities as a function of various monomer concentrations

of cellulose due to a lower quantity of solvent, which inhibits the monomer molecules' ability to penetrate the cellulose molecules, resulting in fewer sites that are actively involved in the reaction at the backbone of cellulose, and thus continuously decreases the number of reacting sites with a lower level of MeOH present in the case of upper monomer concentration. It is evident that the rate of cross-linking formation was proportional when radiation was initially applied. However, the termination of radical-radical reaction was enhanced greatly by an increase in the monomer concentration and consequently reduced the degree of scission reaction and oxidation [29, 30].

3.2 Effect of UV radiation on the mechanical performance of the composites

Strength is a vital physical property of any textile material, since every change in physical or chemical composition always results in a variation in strength. That also happened in our case. The evaluation of the tensile strength (TS), Young's modulus (YM), flexural strength (FS), flexural modulus (FM) and impact strength (IS) of virgin PALF/PLA composites were expressed by taking the mean values that were determined to be 111.7 ± 3.5 MPa, 10.8 ± 1.1 GPa, 175 ± 6 MPa, 13.6 ± 0.7 GPa, and 15.3 ± 0.8 KJ/m^2 respectively (according to Table 4). It is evident from the table that the tensile and flexural properties of the biocomposite are higher than neat PLA after the incorporation of PALF, as anticipated. The graphical presentation of the TS, YM, FS, FM and IS of monomer (HEMA, and MMA) treated PALF composites are seen in Figures 5-7 against the intensities of UV with regard to monomer concentration. The highest TS of 156.3 ± 3 MPa, YM of 15.4 ± 0.15 GPa, FS of 243 ± 5 MPa, FM of 18.7 ± 0.8 GPa, and IS of 21.6 ± 0.9 KJ/m^2 were achieved by the HC sample (30% HEMA) while the optimum TS of 149.4 ± 4 MPa, YM of 14.9 ± 0.3 GPa, FS of 237.3 ± 5.5 MPa, FM of 18.5 ± 0.7 GPa and IS of 20.9 ± 0.6 KJ/m^2 were achieved by the M_4 (40% MMA) sample respectively. Analysed data showed that TS, YM, FS, FM, and IS improved to 40, 42.6, 38.9, 37.5 and 41.7% respectively for the HC composite, while the results for the MC composite were 33.7, 38.4, 35.6, 36.2 and 37.2% respectively compared to the UC sample. The values indicate that the amount of grafting directly influences mechanical properties. The higher the grafting, the better the mechanical behaviours of the composites. Experimental results also showed that TS, YM, FS, FM and IS rise with an increase in UV intensity up to a certain dose and then declined as UV intensity rose. It was reported that maximum TS, YM, FS, FM and IS were exhibited by the HC sample at the 15th UV pass, and at the 20th UV pass for the MC sample. The reason for the improvement in mechanical properties with an increase in UV doses may be attributed to intercross-linking formation among adjacent

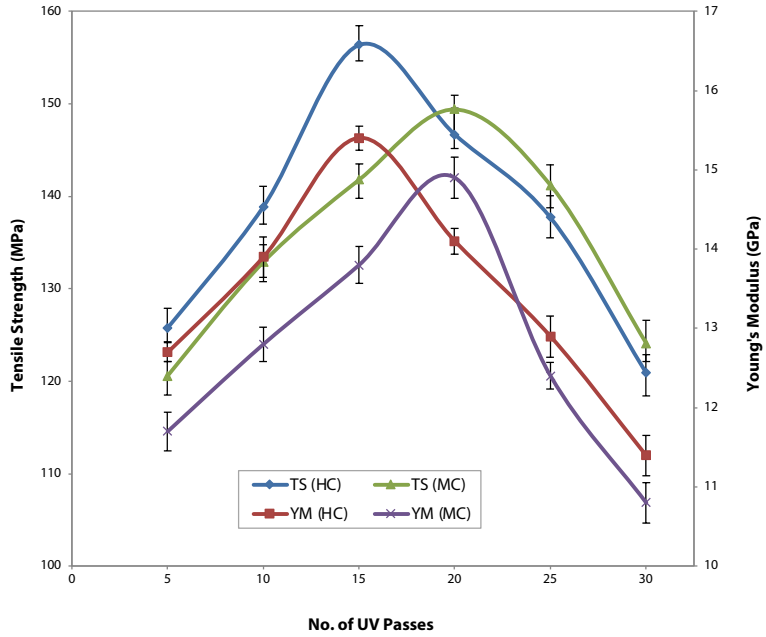


Figure 5: Tensile strength and Young's modulus of monomer treated optimized PALF/PLA composites with regard to UV intensities

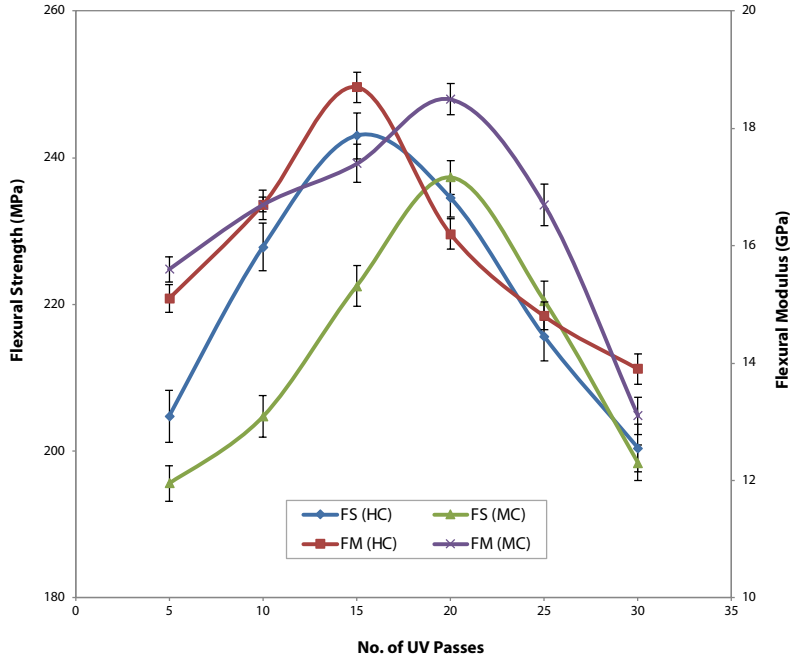


Figure 6: Flexural strength and Flexural modulus of monomer treated optimized PALF/PLA composites with regard to UV intensities

cellulose elements. During the application of UV light, free radicals were created by the photoinitiator (Darocur-1664) and are responsible for starting the free radical reaction. Originally, free radical reactions take place among -OH groups existing in cellulose and monomers and consequently develop properties. During the reaction process, inter reaction between -OH groups may also occur. In the case of HEMA, the reaction mechanism comprises two stages: first, the formation of poly(HEMA) and second, the acrylic groups present in HEMA react with the –OH group of PALF, as illustrated in Figure 8 (a) and 8 (b) respectively. Similar mechanisms also occurred between MMA and PALF during UV curing.

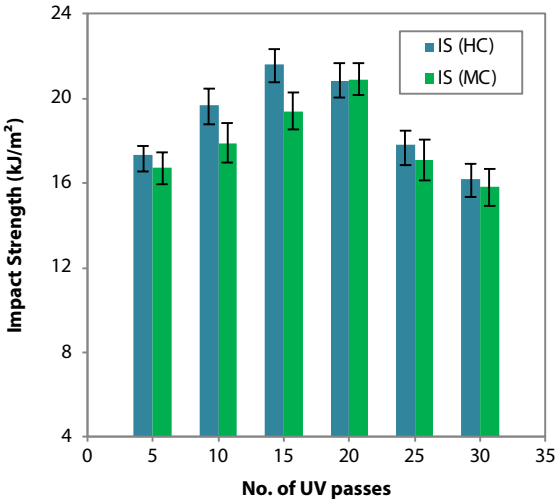


Figure 7: Impact strength of monomer treated optimized PALF/PLA composites with regard to UV intensities

Fundamentally, initiators assist in the initiation of the reaction of the monomer, through which free radical oxygen is formed, but does not actually impart in the reaction. At this time, a homo-polymerization reaction may occur. The treatment of cellulosic fibres with monomers reduces the hydrophilic property, which also imparts developed tensile properties. The experimental data showed that under UV radiation, the mechanical properties improve to a guaranteed value, after which declines may be attributed to two contrasting and simultaneous occurrences referred to as photo-crosslinking, which is responsible for the development of fibre properties and photo-degradation, for which reason fibre characteristics deteriorated. In the case of lower intensities, photo-crosslinking takes place due to the stabilization of free radicals by combination reaction. The grafting efficiency is higher if a higher number of active sites is created on the polymer. At higher intensities of radiation, however, polymer degrade into fragments due to the breaking of the main chain, resulting poor mechanical properties [31, 32].

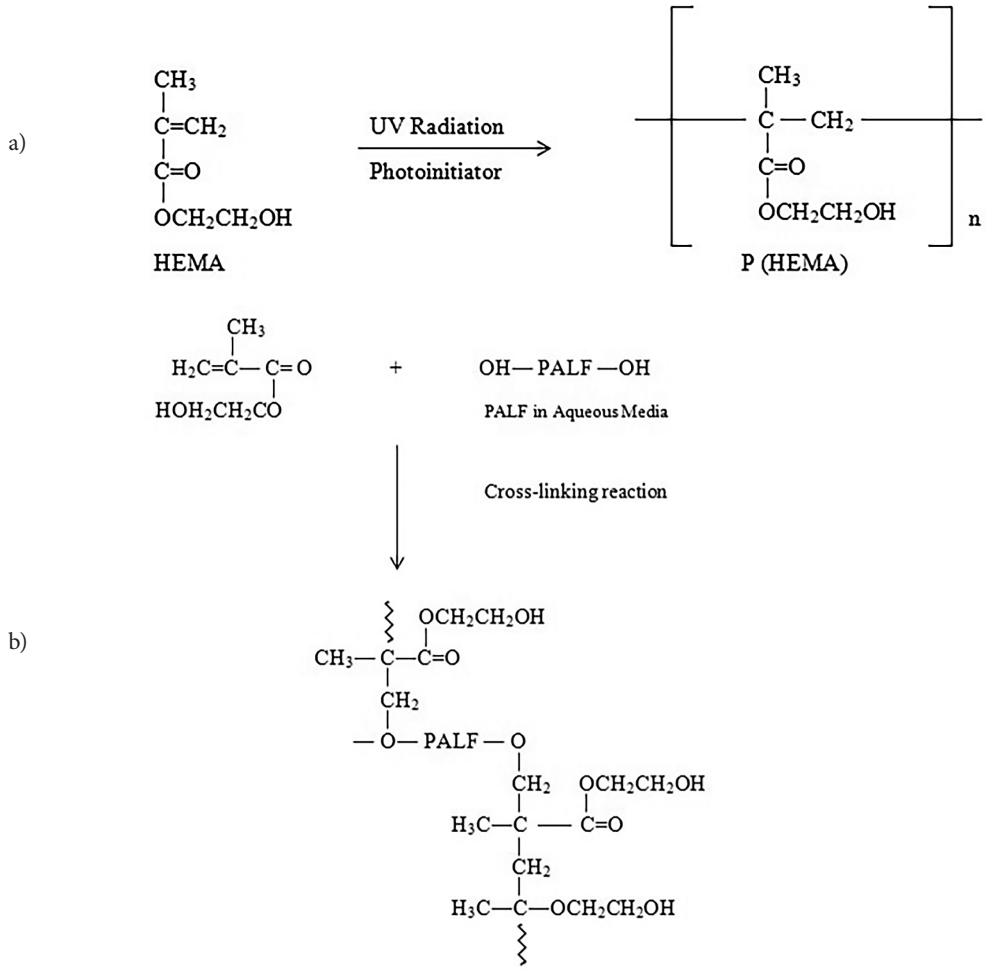


Figure 8: a) Formation of poly(HEMA) during applying UV radiation; b) Cross-linking mechanism between PALF and HEMA

The improvement in the mechanical properties of composites due to the incorporation of photografted PALF is also evident from the stress-strain curve. Typical tensile stress-strain curves of the neat PLA matrix, and the untreated PALF/PLA and monomer treated PALF/PLA composites are shown in Figure 9. In the figure, neat PLA shows a more linear behaviour, while the composites behave more non-linearly as the strain increases. The linear phase corresponds to the linear deformation of the fibre and matrix, while the nonlinear deformation of the composites has been explained as a three-phase mechanism. First, a microcrack initiates at the fibre-end/matrix interface and propagates along the fibre lengths. Second, the matrix undergoes plastic deformation. Finally, the microcracks in the matrix open and propagate through the deformed matrix. Due to the pulling out of fibres from the matrix, catastrophic crack propagation also takes place through the matrix.

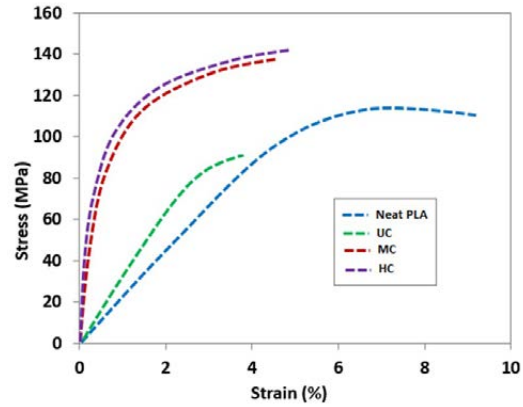


Figure 9: Tensile stress-strain curve of neat PLA and PALF/PLA composites

3.3 Analysis of the mechanical behaviours of composites following the integration of an additive

Experimental data revealed that the H_3 (30% HEMA) and M_4 (40% MMA) solutions provided improved properties of the treated PALFs. During treatment, various concentrations (0.5–1.5%) of urea were incorporated into the augmented formulations. Tables 5 and 6 show the achieved results of TS, YM, FS, FM and IS with G_f (%). The G_f and mechanical behaviours improved due to the addition of urea into the H_3, and M_4 solutions at optimal radiation intensity. The best enhanced mechanical properties were achieved through the addition of 1% urea. It was reported that HC sample (30% HEMA treated + 1% urea) showed an increase of 51% in TS, 49.5% in YM, 45.7% in FS, 46.3% in FM and 47.4% in IS compared to the UC sample. A similar increase was also observed for other monomer treated composites by adding urea. The presence of >C=O groups neighbouring the nitrogen atom in urea has an elongated pair of electrons; by activating them during reaction through an additive, a bridge is formed between cellulose and monomer. Oxygen existing in >C=O groups has a great affinity to electrons. As a result, electrons are closely populous around it, thus drawing additional electrons from the nitrogen atom of urea, and generating certain advantageous situations for the expansion of the monomer molecules and the backbone of cellulose through additives. Urea possesses some properties that when added would stimulate segregation by complex compound formation with monomer molecules, which might lead to an increase in the concentration of monomer at the grafting position and thus accelerate the reaction mechanism at that point [33].

3.4 Thermogravimetric analysis (TGA)

One of the restrictive features in using plant-based lignocellulosic fibres in composite materials is lower thermal stability. In this study, an attempt was made to increase the thermal stability of PALF/PLA composites through photografting. The thermal stability of the UC, HC, and MC samples were studied using TG and DTG curves, as illustrated in Figures 10 and 11 respectively. Both untreated and treated fibre composites lost weight in three steps, although decomposition actually occurs in two main phases that are similar to other lignocellulosic fibre composites, as shown in TG curves. For the UC (virgin PALF/PLA) composite, initial weight loss (of 9.9%) was recorded at between 30–105 °C due to the removal of moisture from the fibres. At temperatures above 200 °C (onset temperature), however, thermal stability was reduced and fibre degradation occurred. In brief, stage I (200–285 °C) corresponds to the cleavage of glycosidic linkages of cellulose with the thermal degradation of hemicellulose and pectin (18.6% weight loss), while stage II (275–382 °C), where maximum weight loss (53.5%) was seen, corresponds to α-cellulose degradation. As lignin consists of aromatic rings that make it complex, its structure decomposed slowly over the entire range of temperatures [34]. Table 7 shows the weight loss (%) of untreated and monomer grafted PALF/PLA composites at different temperature intervals.

Table 5: Grafting and tensile properties of photografted PALF/PLA composites treated with urea

Composites	Concentration of urea								
	0.5%			1%			1.5%		
	G_f	TS (MPa)	YM (GPa)	G_f	TS (MPa)	YM (GPa)	G_f	TS (MPa)	YM (GPa)
HC	18.6	162.4 ± 3.2	15.8 ± 0.14	19.7	168.6 ± 3	16.1 ± 0.57	18.9	163.1 ± 2.8	15.9 ± 0.16
MC	16.5	155.3 ± 2.8	15.3 ± 0.25	17.4	161.2 ± 2.4	15.7 ± 0.22	16.3	154.8 ± 2.5	15.2 ± 0.27

Table 6: Flexural and impact properties of photografted PALF/PLA composites treated with urea

Composites	Concentration of urea								
	0.5%			1%			1.5%		
	FS (MPa)	FM (GPa)	IS (kJ/m^2)	FS (MPa)	FM (GPa)	IS (kJ/m^2)	FS (MPa)	TS (MPa)	IS (kJ/m^2)
HC	249.3 ±5	19.3 ± 3.2	21.4 ± 0.7	255.1 ±6	19.8 ± 3	22.5 ± 0.3	250.2 ±4	19.4 ± 1.8	21.6 ± 0.5
MC	243.8 ±4	18.9 ±2.8	20.6 ± 0.3	251.4 ±5	19.3 ± 2.4	21.7 ± 0.2	245.3 ±6	18.8 ± 1.7	20.7 ± 0.27

Table 7: TG data of untreated and grafted PALF/PLA composites

Composites	Temperature (°C)			Weight loss (%)		
	Initial stage	Stage I	Stage II	Initial stage	Stage I	Stage II
UC	30–105	200–285	285–398	9.9	18.6	66.7
HC	90–198	251–343	343–492	4.6	8.7	58.8
MC	82–192	245–332	332–479	4.5	9.1	59.6

It can be clearly seen from the graphs that grafting increased the thermal stability of PALF/PLA composites. All the monomer treated grafted fibre composites showed improved thermal stability compared to that of untreated samples, as presented in Figure 10. In the initial stage, treated fibre composites (both HC and MC) demonstrated very low weight loss (5.3 and 5.8% for the HC and MC sample respectively), which actually depends on the amount of grafting, as well as the small amount moisture present in the grafted fibre. Grafted composite samples also exhibited a significantly lower amount of weight loss in stage I (8.7–9.1% between 245–343 °C) and stage II (58.8–59.6% between 332–492°C) than the untreated samples. Among the treated samples, 30% HEMA treated PALF/PLA (HC) we more resistant to heat than other samples, probably due to their stronger cross-linkage formation than the other samples. The reaction between the chemical components of monomers and –OH of PALF changes the structure by forming cross-linkage, resulting in the improved strength of the mechanical bond between PALF and the monomer. The complex structure of cross-linkage prevents further degradation, which in turn increases the degradation temperature in stages I and II, and is also responsible for the low weight loss in these two stages. Moreover, free radicals formed due to UV radiation also react with the cellulose and alter its chemical nature, thus creating more hydrophobic and stronger covalent bonds, and increased thermal stability [35]. The degradation of PALF is primarily dependent on the degradation of cellulose, which is the leading constituent of the fibre. However, the chemical structure of cellulose is altered due to photografting, which influences the degradation reaction of cellulose and thus the degradation temperature. Together, this contributes to the enhanced thermal stability of photocured PALF/PLA composites. DTG curves (Figure 11) also provide evidence of the three-stage degradation of modified fibre composites, with weight loss profiles that confirm the developed thermal properties of the monomer treated PALF/PLA.

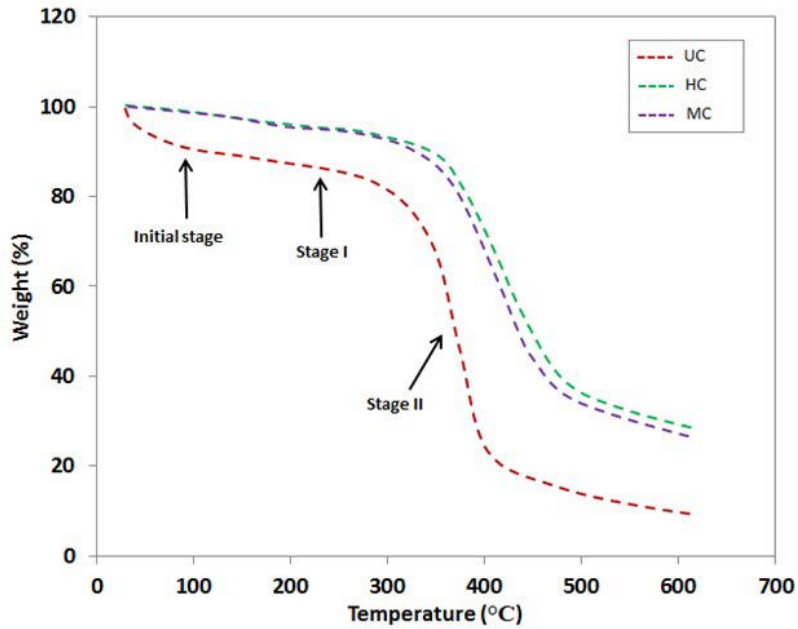


Figure 10: TG curves for untreated and photografted optimized PALF/PLA composites

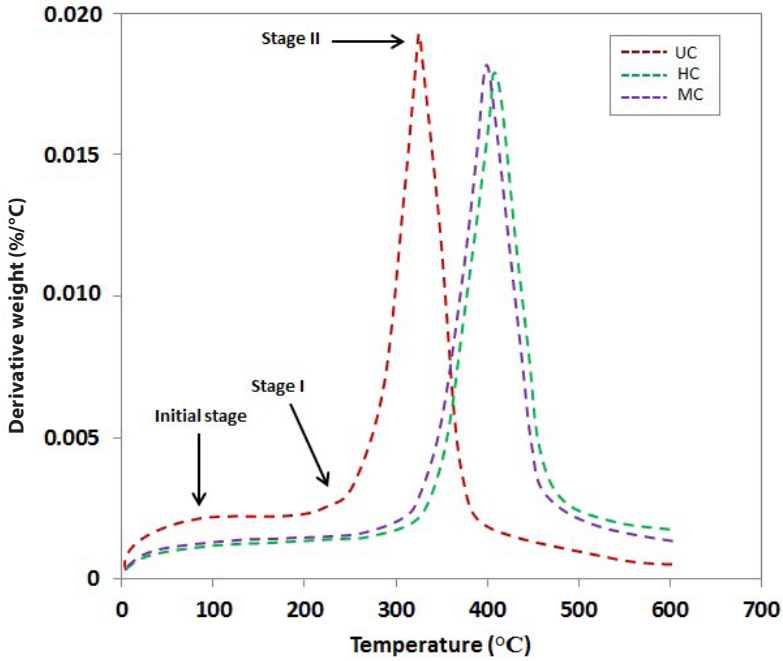


Figure 11: DTG curves for untreated and photografted optimized PALF/PLA composites

3.5 Differential scanning calorimetry

The melting behaviour and degree of crystallinity of neat PLA and PALF/PLA composites were examined using DSC. Figure 12 shows the DSC curves of the UC, HC and MC samples, together with pure PLA, and a summary of results is presented in Table 8. It is evident from the graphs that the addition of PALF fibre into the PLA matrix changed the glass transition and melting temperature of the composites. Observations reveal that the untreated fibre composite exhibited improved T_g relative to neat PLA, which indicates the changing properties from flexible to hard [11]. It is evident that the melting temperature (onset) and melting enthalpy decreased marginally due to the incorporation of PALF into PLA, which may be attributed to the role of PALF as a diluent, resulting in the smaller amount of heat required to melt the UC and the resulting lower T_m. Another reason may be that the polymer chains in PLA might be diffused and weakened due to the presence of PALF [10]. Due to radiation induced grafting, the hydrophilic characteristic of PALF is greatly reduced, which further develops adhesion between PALF and PLA resulting in the higher melting temperature of photocured PALF composites (HC and MC) than the untreated sample (UC).

It is evident from Table 8 that crystallinity (%) decreases when PALF is added to the matrix PLA, which is due to the amorphousness of PALF. Nevertheless, (monomer + UV) treated composites demonstrated an increased degree of crystallinity because, during grafting under radiation treatment, both fibre and monomer have active points and produce a complex structure by forming cross-linkage, which ultimately reduces amorphousness and increases crystallinity (%).

Table 8: Thermal properties of neat PLA and grafted PALF/PLA composites

Material	Glass transition temperature, T_g (°C)	Melting temperature, T_m (°C)		Melting enthalpy, ΔH_m (J/g)	Degree of crystallinity, X_c (%)
		Onset melting temperature	Peak melting temperature		
Neat PLA	61	167	175	46	49
UC	63	151	162	32	34
HC	65	161	169	37	39
MC	64	157	166	36	38

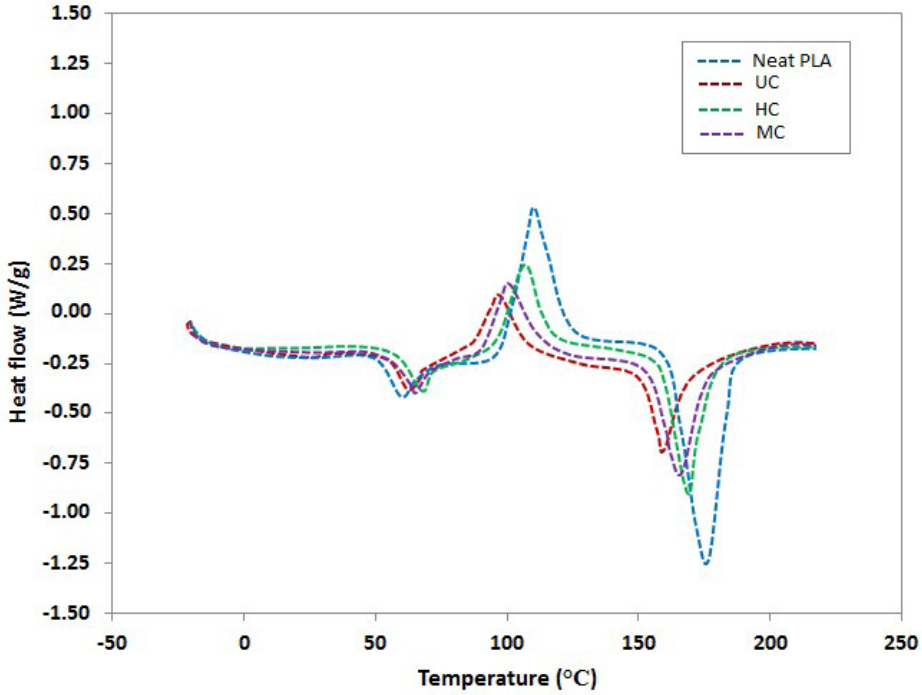


Figure 12: DSC curves for untreated and photografted optimized PALF/PLA composites

3.6 Swelling behaviour

Water absorption indicates the soaking performance of fibres, which is deemed to be a barrier property. Figure 13 shows water absorption (%) with respect to swelling time (days). For this test, neat PLA, UC (composite without treatment of PALF), HC (30% HEMA treated at the 15th UV pass) and MC (40% MMA treated at the 20th UV pass) samples were selected. Observations reveal that the UC sample soaks water in a distinctive way, while the monomer treated samples absorb water at very high rate during the initial 10–15 days. After that time, the absorption rate declined in an almost static manner, while the untreated fibre composite demonstrated continuous water soaking with the progression of time. After 30 days, neat PLA and UC sample absorbed water up to 2.5, and 75.8% respectively, while that rate was 38 and 40.6% for the HC and MC samples respectively, which is actually determined by their grafting values. Because of grafting, void space in the fibre was filled by polymers, and several hydroxyl groups present in the cell wall polymer were replaced during the bond formation of chemical groups, thus reducing the hygroscopicity of treated fibre. The degree of crystallinity of the composites is also responsible for their water retention behaviours, as pure PLA demonstrated a degree of crystallinity of 49%, which decreased to 39, 38 and 34% for the HC, MC and UC composites respectively. It was thus established that water absorption primarily occurred in amorphous regions. For this reason, grafted fibre composites exhibited lower water absorption than untreated composites. Thus, from the above result, it can be stated that an increase in grafting values significantly reduced the water retention capacity of photografted fibre reinforced composites.

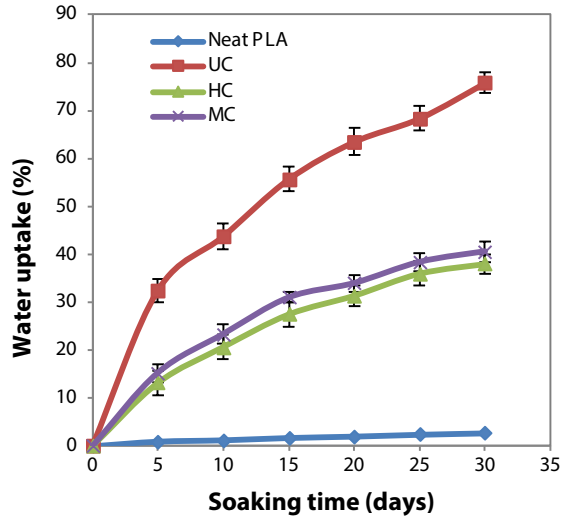
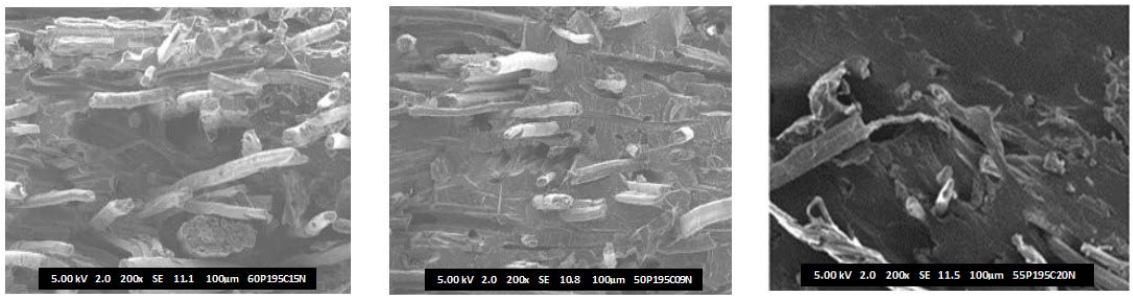


Figure 13: Water uptake (%) of untreated and monomer treated optimized PALF/PLA composites with regard to soaking time



a) b) c)

Figure 14: SEM micrograph of: a) untreated PALF/PLA composite, b) and c) 30% HEMA treated PALF/PLA composite

3.7 SEM analysis

To understand fibre matrix adhesion, SEM micrographs of the composites were studied. The tensile fracture surface of untreated and monomer treated PALF/PLA composites are shown in Figures 14 (a), and 14 (b) and (c) respectively. The untreated PALF partially adhered to the matrix PLA, indicating weak adhesion. More voids and debonding were found on untreated the PALF/PLA composite. A high degree of fibre agglomeration and wider gaps at interfaces result in poor mechanical and thermal properties. An improved fibre-matrix bonding in grafted PALF/PLA was seen, as PALF was entirely surrounded with PLA, while good dispersion can be seen in Figure 14 (c). Also evident is a lower number of voids with improved fibre distribution, indicating a better interface between fibre and matrix, which in turn led to enhanced mechanical and thermal properties, which supports the achieved experimental results.

4 Conclusion

A photografting technique was used to modify PALF fibres with two types of vinyl monomers, i.e. HEMA and MMA, while the mechanical properties of produced composites were successfully assessed. Based on grafting and mechanical properties, the concentration of monomers and radiation intensity were augmented. Taking into account the relevant parameters, the achieved results illustrated that composites made of PALF grafted with 30% HEMA at the 15th UV pass and 40% MMA at the 20th UV pass of UV radiation resulted in optimized mechanical properties. Moreover, the addition of urea (1%) into the optimized solution significantly enhanced the mechanical properties of the composites. Optimized mechanical properties were achieved by fragmenting (glucosidic + weaker) bonds and forming a stronger cross-linkage. The water uptake behaviour of the grafted sample showed a more hydrophobic nature than the virgin sample. Thermogravimetric studies demonstrated that photografting improved the thermal stability of the composites, as well as their resistance to degradation under heat. Although various surface pretreatments can improve the mechanical properties of cellulosic fibre, it can be concluded with a high degree of certainty from this experimental study that photografting is an effective, safe and pollution-free process for the development of the thermo-mechanical behaviours of PALF/PLA composites, which can lead to prospects for the commercial and industrial application of PALF fibres.

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Synthesis and Characterisation of Azo-Based Dichlorotriazine Reactive Dye with Halochromic Behaviour

Sinteza in karakterizacija diklorotriazinskega reakcijskega barvila na azoosnovi s halokromnim odzivom

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Abstract

Halochromism or pH sensitivity has tremendous potential for applications in various textile fields, such as protective clothing, wound dressings, etc. Reactive dye is mostly used to colour cotton or other regenerated cellulose fibres due to its better fastness and wide range of hue, from vivid to dull shades. In this research work, an azo-based dichlorotriazine reactive dye was synthesised from H-acid (4-amino-5-hydroxy-2,7-naphthalen-edisulfonic acid) and 4-nitroaniline, which incorporates a halochromic behaviour. The change of colour of this dye was evaluated both in the the solution stage and coloured fabric stage in various pH solutions. A visible change of colour with the alteration of pH was observed after dyeing textile fabric with the synthesised dye. However, a significant difference was observed in a few cases with regard to the change of colour with the alteration of pH in the solution stage and coloured fabric stage. The dyed fabric also displayed very good to excellent wash fastness properties. Generally, the reactive dye synthesised in this research demonstrated an obvious change of colour with the alteration of the pH level.
Keywords: chromism, H-acid, 4-nitroaniline, synthesis, halochromic, dichlorotriazine, reactive dye

Izvleček

Halokromizem ali občutljivost na pH ima široke možnosti za uporabo na različnih tekstilnih področjih, kot so zaščitna oblačila, povoji za rane itd. Reaktivna barvila zaradi boljše obstojnosti in širokega razpona barvnih odtenkov, od živih do pastelnih tonov, večinoma uporabljajo za barvanje bombaža ali regeneriranih celuloznih vlaken. V tej raziskavi je bilo iz 4-amino-5-hidroksi-2,7-naftalendisulfonske kisline (H-kislina) in 4-nitroanilina sintetizirano diklortriazinsko reaktivno barvilo na azoosnovi. Barvilo omogoča halokromni odziv, to je spremembo barve, ki je bila ocenjena v raztopini in na obarvani tekstiliji pri različnih vrednostih pH raztopin. Po barvanju tkanine s sintetiziranim barvilom je bila opažena vidna sprememba barve s spreminjanjem vrednosti pH. V nekaj primerih je bila ugotovljena tudi pomembna razlika

v spremembi barve s spremembo vrednosti pH raztopine in na obarvani tkanini. Obarvana tkanina je imela tudi zelo dobro do odlično obstojnost pri pranju. Na splošno je reaktivno barvilo, sintetizirano v tej raziskavi, pokazalo zaznavno spremembo barve s spreminjanjem vrednosti pH.
Ključne besede: kromizem, H-kislina, 4-nitroanilin, sinteza, halokromen, diklorotriazin, reaktivno barvilo

1 Introduction

Chromic materials reversibly change colour as a reaction to a change in external stimulus. Based on the nature of the stimulus, the resulting chromism is classified as thermochromism (induced by temperature), photochromism (induced by light), halochromism (induced by pH), etc. [1]. Chromic materials are widely used to cover products to exhibit chromic phenomena in the field of smart or intelligent materials [2]. For various textile uses, such as wound dressings, protective garments, etc., the concept of halochromism may be of tremendous significance. Because of their flexibility, comfort, and ability to cover huge surfaces, these halochromic textile pH-indicators are more beneficial than traditionally applied sensor elements. For this reason, research usually focuses on the connection between the chemical construction of dyes and their halochromic nature in liquid instead of their performance in a textile substance. The performance evaluation of the dyes absorbed in a textile material is also essential [3].

Azo dyes have been well-accepted on the global market because of their simple synthesis process, the capability to produce a wide range of shades, and their vast applications in various fields, such as papers, textiles, additives, cosmetics and organic synthesis [4–5]. Around 60–70% of azo dye is consumed in the traditional textile wet-processing industry [6]. In the case of azo dye, an associated π-configuration is observed, and is linked to two aromatic subdivisions (generally benzene and naphthalene derivatives) by a nitrogen–nitrogen double bond (N=N) [7–8]. As a result of the fairly easy synthesis process and the wide range of colours, azo dyes are the most available category of colourants. Some azo dyes change colour because of their chromic characteristics related to extrinsic consequence [2]. Reactive dye has the ability to remove the unfixed dye from the fabric surface more efficiently, ensuring excellent washing fastness to dyed or printed fabric based on the colour change in shade and staining of an adjacent textile substrate [9]. Reactive dyes have the advantage over natural dyes and other synthetic dyes. They form a covalent bond between the dye and fibre after application to the fibre surface. This covalent bond is produced between a carbon atom of the dye molecule and an oxygen, sulphur, or nitrogen atom of an amino, hydroxy, or thiol compound on the polymer matrix. Due to the formation of a strong covalent bond after the application of dyes to the textile substrate, this dye is difficult to remove and, as a result, has superior wash fastness characteristics [6].

Today, several examples of chromic phenomena have been applied in our everyday life, such as photochromic lenses for spectacles and thermochromic temperature indicators, for example, in baby spoons [10]. Another significant aspect of chromism is halochromism, which depends on the degree of acidity or pH level. Halochromic or pH-sensitive materials, included under the subclass of ionochromic materials, vary in colour depending on pH, and are currently subject to more frequent researches [11]. Halochromic dyes that cause a change from one colour to another due to a bathochromic or hypsochromic shift of the absorption peak upon (de)protonation are categorised as positive and negative halochromic dyes, respectively [12]. The basic principle of the colour change of a halochromic substance is the protonation or deprotonation of the dye molecule, causing a different electron arrangement, resulting in a change in the colour of that material. The origin of apparent colour alteration of halochromic dye is the ring-opening of the dye molecule upon (de)protonation, or on tautomerism, as tautomers have various colours and tinctorial strengths [13]. In addition to the more well-known thermochromism and photochromism, the comparatively less-utilised halochromism or pH sensitivity has tremendous potential for use in various textile applications as pH sensors [3]. Halochromic textiles have huge potential as sensors that can signal a medium's pH through the simple visual observation of colour, and can be implemented in a wide variety of fields [14]. Hesus et al. (1892) first declared that the human skin surface is acidic, and this concept was confirmed again by Schade and Marchionini (1928) [15, 16]. This acidic medium differs in the pH range between 4–6, which is influenced by a person's anatomical state and age. Due to the presence of different acids, such as amino acids, fatty acids and others

formed and discharged by the keratinocyte coating and the skin protuberances, the natural lactate-bicarbonate buffer structure of the body shifts on to an acidic medium [16]. In chronic wounds, the medium of pH has been found in the range of 7.15–8.9. A wound's pH shifts alkaline to neutral and later becomes acidic as the wound advances towards the healing process [17]. A halochromic wound dressing can be implemented as an indicator to monitor the state of the wound surface's healing just by observing the alteration of colour in the bandage without removing the gauze. Thus, a pH-indicator wound dressing can be an effective observation system.

Consequently, potential damage to a patient's wound area might be reduced, as well [14]. For this reason, the successful application of halochromic dye as a textile pH indicator on wound dressing can play a crucial role in the field of medical textiles. Furthermore, in the human body, pH value plays a significant role in many other crucial processes. Thus, halochromic textile might play a vital role in determining the acidity of sweat, nasal discharge, urine, etc. [18, 19]. On the other hand, a textile pH indicator in protective clothing can identify the existence of acid vapours in an operating environment [1]. However, no significant previous research attempt or literature has been found to synthesise reactive azo-based halochromic dye as a potential pH sensor for textile materials.

The principal aim of this research article was to synthesise a new azo-based dichlorotriazine reactive dye from H-acid (4-amino-5-hydroxy-2,7-naphthalenedisulfonic acid) and 4-nitroaniline, which have halochromic properties. The change in colour of this dye was observed in various pH solutions. After that, 100% cotton woven fabric was dyed with the newly synthesised dye following the conventional dichlorotriazine reactive dyeing procedure. In addition, the halochromic behaviour of the dyed fabric was investigated at various buffer pH solutions. To investigate and analyse the halochromic behaviour of synthesised dye in the solution stage, analytical methods such as UV-vis spectroscopy, were used. CIELAB values of the dyed fabric in various pH solutions were examined using a chroma meter. Finally, the purity and molecular arrangement of the synthesised dye were investigated using thin-layer chromatography (TLC).

2 Experimental

2.1 Fabric

In this research, 100% cotton plain weave fabric, with a mass of 102.93 gm/m^2, and a fabric density of 48 threads/cm in warp and 32 threads/cm in the weft direction was used. The fabric was provided by Whaley's of Bradford (UK).

2.2 Chemicals

For dye synthesis, H-acid (4-Amino-5-hydroxy-2,7-naphthalenedisulfonic acid, 88%), cyanuric chloride (≥ 99%), 4-nitroaniline (≥ 99%), nitrous acid (HNO_2) (99%), sulfamic acid (H_3NSO_3) (99%), dihydrogen phosphate ($[H_2PO_4]^-$) (≥ 99%) and disodium hydrogen phosphate (Na_2HPO_4) (≥ 99%) were supplied by Sigma-Aldrich, UK and used as received. Disodium hydrogen phosphate (Na_2HPO_4) and citric acid ($C_6H_8O_7$) were used for the preparation of buffers.

2.3 Synthesis of azo-based dichlorotriazine reactive dye

The synthesis of the dye comprised three steps: modification of H-acid (4-amino-5-hydroxy-2,7-naphthalenedisulfonic acid), diazotising of nitroaniline (4-nitroaniline) to obtain diazonium salt, and coupling by reacting the obtained diazonium salt in the modified H-acid (4-amino-5-hydroxy-2,7-naphthalenedisulfonic acid) solution.

Modification of H-acid (Step 1)

H-acid (4-Amino-5-hydroxy-2,7-naphthalenedisulfonic acid, 5.0 g, 0.012 mol) was dissolved in

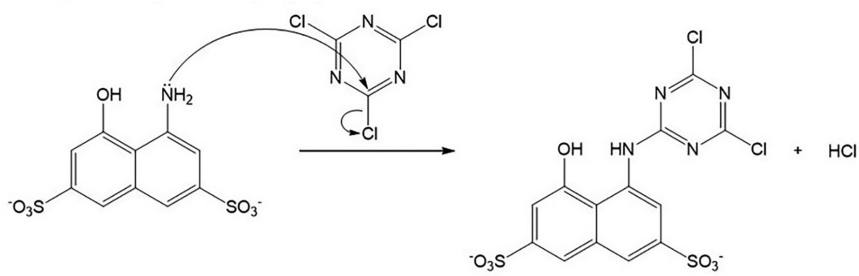

Figure 1: Route for the formation of dichlorotriazine reactive dye

50 cm³ water and the pH adjusted to pH 7 with an NaOH dilute. The solution was then cooled to 0–5 °C in an ice bath. Cyanuric chloride (2.8 g, 0.015 mol) was dissolved in acetone (20 cm³), cooled in an ice bath to 0–5 °C, then added to the H-acid solution over 30 minutes at 0–5 °C with no pH control. The reaction, shown in Figure 1, was stirred for a further 15 minutes, monitored at intervals for the presence of free aromatic amine with Ehrlich reagent. Small amounts of cyanuric chloride dissolved in acetone were added at 15-minute intervals, if required, to lead the reaction to completion, after which the pH was raised to pH 6 using NaOH (1M).

Diazotisation (Step 2)

4-nitroaniline (1.65 g of nitroaniline; 2.24 g of dinitroaniline, 0.012 mol) was dissolved in water (50 cm³) and cooled to 0–5 °C in an ice bath. Concentrated hydrochloric acid (3 cm³) was then added with mechanical stirring. A solution of sodium nitrite (1.0 g, 0.014 mol) in water (20 cm³) was added to the solution of nitroaniline in drop form over 15 minutes. When the addition of sodium nitrite was complete, the reaction solution was stirred for a further 30 minutes at 0–5 °C. A small quantity of sulfamic acid (99%, 97.09 g/mol) was added to destroy excess nitrous acid until starch–iodide paper no longer turned blue.

Coupling (Step 3)

The modified H-acid solution of step 1 was cooled to 0–5 °C, and its pH raised to pH 9–10 with the addition of NaOH. The diazonium salt prepared in step 2 was poured into an ice-cold solution containing 7.4 g sodium acetate and 3.63 g acetic acid to raise its pH to a neutral level. The diazonium salt solution was immediately added to the modified H-acid from step 1 in drop form over 30 minutes at 0–5 °C. The pH of the solution was kept between 9.5 and 10, using NaOH dilute throughout the process. The solution was then left stirring for one hour, after which the temperature was no longer controlled.

Precipitation and filtration procedure of the synthesised dye

After completing the dye synthesis, sodium chloride (20% w/v) was added to the synthesised dye solution to separate the dye. The solution was stirred continuously using a mechanical stirrer until all of sodium chloride was dissolved. The separation of the solid dye from the liquid mixture was achieved using vacuum filtration. In a Buchner funnel, the combination of precipitated dye and fluid was discharged over a filter paper. The liquor was drained over the funnel into a flask through a vacuum, and filter paper captured the solid dye. Finally, the filtrated dye was mixed with 0.6 g potassium dihydrogen phosphate and 1.4 g disodium hydrogen phosphate. The dye was then placed in a desiccator for 12 hours to dry thoroughly.

2.4 Dyeing procedure of synthesised dichlorotriazine reactive dye

The dyeing procedure, schematically presented in Figure 2, was carried out in Labomat BFA-8 (Mathis, Switzerland) infrared lab dyeing machine. Cotton fabric was dyed using the dichlorotriazine reactive dyeing technique. A higher dye concentration (5% on the mass of fabric) was applied to get a reasonably deep shade. With specific concentrations of dyestuff, 60 g/L of salt and 10 g/L of soda ash were applied for the dyeing of cotton fabric with synthesised dye. The liquor-to-goods ratio used in this dyeing process was 10:1. At first, the required amount of dyestuff was added to a dye bath at at a temperature of 30 °C. The salt was then added in three steps at intervals of 10 minutes, 10 minutes and 15 minutes. The dye bath was run for 15 minutes in this condition. After that, soda ash was added in two steps, at 15-minute intervals. The fabric was dyed for 30–40 minutes at 30 °C [20].

After completing the dyeing process, the fabric was rinsed in cold water for 2 minutes, boiled at 80 °C for 5 minutes, warm-rinsed at 60 °C for 5 minutes, and finally rinsed in cold water for 2 minutes. The fabric was then dried thoroughly [20].

2.5 Preparation of McIlvaine buffer system

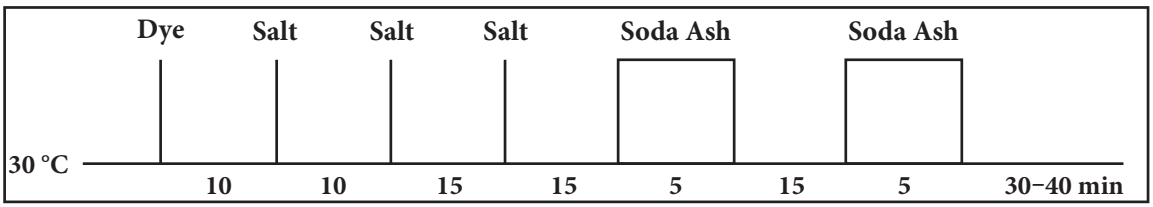

Figure 2: Standard exhaust method for dichlorotriazine-based reactive dye [20]

Table 1: McIlvaine buffer system in different pH [21]

pH required	0.2 M Na_2HPO_4 (cm^3)	0.1 M citric acid (cm^3)
2.2	0.40	19.6
3.0	4.11	15.89
4.0	7.71	12.29
5.0	10.30	9.70
6.0	12.63	7.37
7.0	16.47	3.53
8.0	19.45	0.55

Buffers of different pH values were prepared to measure the change of colour of both dyes in solution and fabric. A total of 20 mL of buffer solution was prepared using 0.2 M disodium hydrogen phosphate and 0.1 M citric acid of the appropriate amount according to Table 1.

2.6 Analysis

UV-vis spectrophotometry

In this study, the change of maximum wavelength and absorbance of dye in different pH solutions was identified using a M550 (CamSpec, UK) double-beam scanning UV/Visible spectrophotometer. All sample solutions were placed in quartz cuvettes with a 10 mm light path. The amount of dye concentration was calculated based on a calibration curve. The spectra were recorded from 400 nm to 700 nm.

Colorimetric measurements

A CS-200 chroma meter (Konica Minolta, Japan) was used to measure the CIELAB values of the dyed fabric to analyse the change of colour in an aqueous solution at different pH values, maintained by hydrochloric acid (HCl) and sodium hydroxide (NaOH). Measurements of colour were performed under the following conditions: white tile: X = 271.04, Y = 281.28, Z = 285.31; viewing angle: 65°; capture angle: 1°.

pH analysis

The pH analysis was performed using a Metrohm 654 digital pH meter (Metrohm, UK) combined with a reference electrode and glass electrode.

Thin-layer chromatography (TLC) analysis

In this research, TLC was used to analyse the purity and individualise the chemical components present in the synthesised dye. Thin-layer chromatography was performed using ethyl acetate-m-butanol-n-propanol-water in a ratio of 1:2:3:4 as the mobile phase, while an aluminium plate coated with silica gel 60 F254 (Merck, UK) was used as the stationary phase. The developed plate was studied under both visible and ultraviolet light (360 nm wavelength), and finally, the retention factor (R_f) was determined [22, 23].

The value of R_f was calculated using the following equation (1):

$$R_f = \frac{\text{distance travelled by the component}}{\text{distance travelled by the solvent}} \quad (1)$$

Colour fastness to washing

The colour fastness to washing test of dyed fabric was carried out according to the ISO 105-CO6:2010 (ISO, 2010) Standard in a GyroWash Tester (James Heal, UK). The washed specimen was evaluated visually using the Grey Scale according to the ISO 105-A02 (for assessing colour change) and ISO 105-A03 (for assessing staining) Standards.

Determination of response time

The response time was recorded after immersing the synthesised dyes into various buffer solutions until a visible change in the colour of the solution occurred. The response time was also determined after absorption of the dyed fabric sample at different buffer solutions until the clear visible alteration of colour in the fabric surface appeared.

3 Results and discussions

The chemical structure of the synthesised dye is shown in Figure 3.

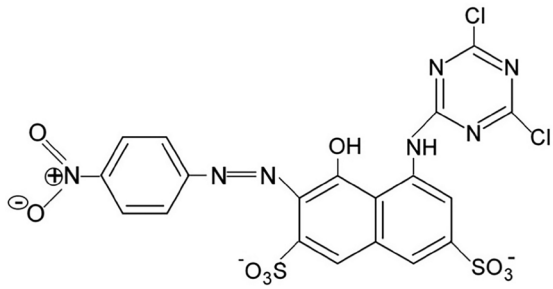

Figure 3: Structure of the synthesised dye

Figure 3 shows that the synthesised dye molecule contains an azo (-N=N-) group as a chromophore or colour- bearing group, and two sulphonate groups (SO_3^-) as a water solubilising group. Due to the presence of two sulphonate groups, the water solubility of this dye is high. The dye molecule possesses a bridging group, such as -NH-, which increases substantivity because of hydrogen bond formation. On the other hand, the dichlorotriazine group acts as the reactive functional system in this synthesised dye [24].

The UV-visible absorption spectrum of the synthesised dye was determined by dissolving it in distilled water (0.05 g/L solution) at a wavelength of between 400–700 nm. From Figure 4, the wavelength of maximum absorption (λ_{max}) of the synthesised dye is identified at 506 nm, which appeared in the visible region of the spectrum due to the ascription on π–π^* transition of the azo (N=N) chromophore group of synthesised reactive dye [25]. The spectral peak appears slightly wide, resulting in some impurities present in the dye [26].

3.1 Visual observation of colour change in the solution stage of dye in different pH solutions

The synthesised dye solution (5 g/L concentration) is applied in different Mcllvaine buffer solutions (pH range of 2.2–12) to detect the visual change of colour, as presented in Figure 5.

It is evident from the visual perception of the dye solution at different pH values shown in Figure 5 that at pH 2.2, the dye's solution appeared reddish-orange. After that, it turned red and continued until pH 7. However, the colour of the solution turned bluish-red at pH 8. The dye solution's colour became purple at pH 9.2 and finally turned dark blue in extremely alkaline conditions at pH 12.

3.2 UV-vis spectrophotometer results of the synthesised dye in different pH solutions

The shifts of maximum wavelength (λ_{max}) and absorbance of the synthesised dye (0.05 g/L concentration) solution were analysed in a buffer solution of different pH values. The results are shown in Table 2.

Table 2 shows that in the visible area of the spectrum, a significant increase of absorbance is detected at 670 nm in pH 7.0. The λ_{max} differs with pH from 622 nm at extremely acidic pH to 430 nm at extremely alkaline pH, which corresponds to the visual colour transition from reddish-orange to dark-blue. The principal reason for the colour change is the protonation or deprotonation of the dye molecules, resulting in various electron configuration changes [27]. Protonation and deprotonation of studied dye are presented in Figure 6.

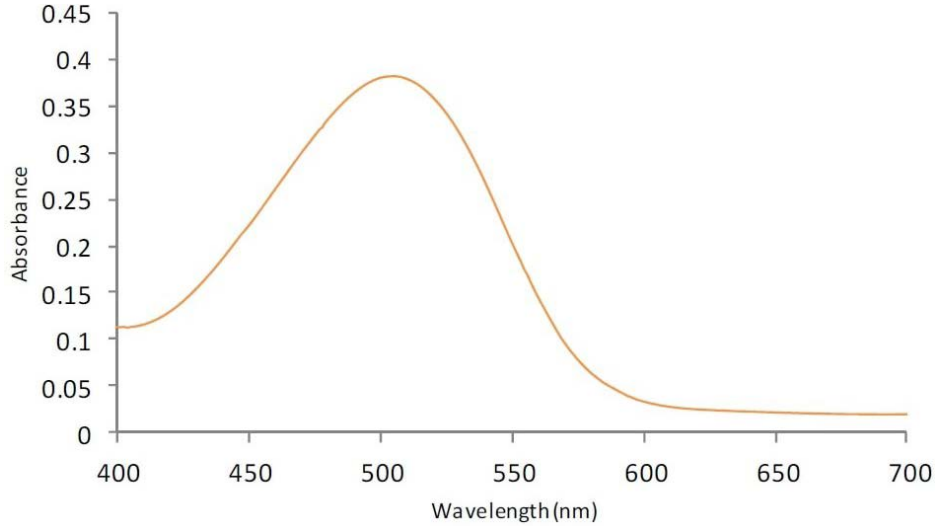

Figure 4: UV-vis spectrum of the synthesized dye

Dye solution pH 2.2
Color: Reddish Orange

Dye solution pH 3
Color: Red

Dye solution pH 4
Color: Red

Dye solution pH 5
Color: Red

Dye solution pH 7
Color: Red

Dye solution pH 8
Color: Bluish Red

Dye solution pH 9.2
Color: Purple

Dye solution pH 12
Color: Dark blue

Figure 5: Colour change of synthesised dye in different pH solutions

Table 2: UV-vis analysis of dye synthesised from H-acid and 4-nitroaniline in different pH solutions

Mcllvaine's buffer pH	Dye solution pH	Maximum wavelength (λ_{max}) (nm)	Absorbance
2.2	2.24	622.00	1.06
3.0	3.02	665.00	1.05
4.0	4.08	667.00	0.75
5.0	4.99	670.00	0.82
7.0	7.01	670.00	1.40
8.0	8.10	630.00	0.95
9.2	9.21	489.00	0.94
12.0	12.11	430.00	0.56

From pH 2.2 to 3.0, a significant bathochromic shift λ_{max} (43 nm) occurred due to the protonation of synthesised dye (Figure 6, structure I_a) [28], while in acidic conditions, the proton and hydrogen bond of the dye's hydroxyl group might be powerful. Therefore, deprotonation becomes more difficult until it reaches acidic to alkaline conditions. The electron-donating substituents, such as hydroxyl (-OH) and amino (-NH-) groups, present in this structure are comparatively less active in acidic and neutral conditions. For this reason, from pH 3.0 to pH 7.0, a negligible bathochromic shift (5nm) was observed. However, these electron-donating substituents gradually became active in alkaline conditions. As a consequence, the dye converted from protonated to deprotonated form (Figure 6, structure II_a). Upon (de)protonation, the dye transformed into anionic form, while the dye's molecular chain became open (Figure 6, Structure II_b), which results in a visible colour change of the synthesised dye. Finally, azo/hydrazone tautomerism was exhibited in the synthesised dye structure (Figure 6, Structure I_b). For this reason, a significant hypsochromic shift λ_{max} (240 nm) was seen with a rise in pH from neutral (pH 7) to extremely alkaline (pH 12) condition [29].

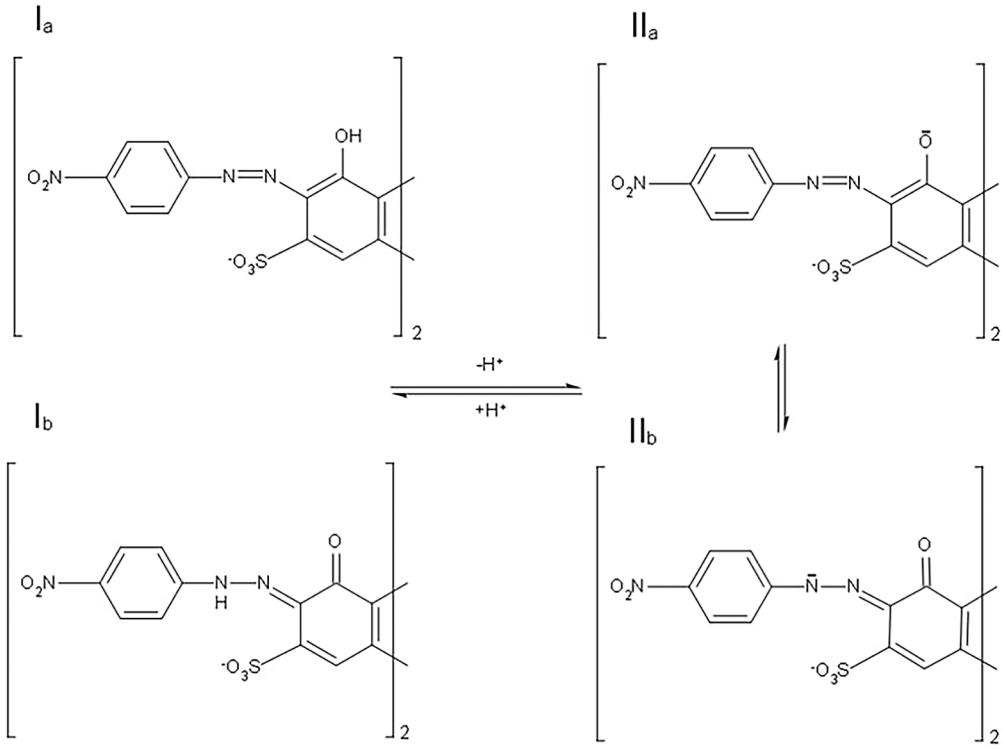

Figure 6: Acid -alkali equilibrium of the synthesised dye in aqueous buffer solutions

3.3 Colour change observation of dyed fabric in different pH solution

The appearance of the cotton fabric dyed with synthesised dye is shown in Figure 6.

The colour of the cotton fabric after dyeing with synthesised dye appeared reddish-pink, as seen in Figure 7. This research further investigated the halochromic behaviour of the dyed fabric sample in different pH solutions, as shown in Figure 8.

Figure 7: Cotton fabric's appearance dyed with synthesised dye concentration of 5% on the mass of fabric (o.m.f.)

It is evident from Figure 8 that an obvious change of colour was observed in the dyed fabric by changing the pH level. In an extremely acidic solution (pH 2.2), the colour of the dyed fabric appeared orange, while at pH 3, the colour became light-orange. The colour of the fabric turned reddish-orange at pH 4. At pH 5, the colour became light-pink, turned in dark-pink at pH 6, and became reddish-pink at pH 7. However, at pH 8, the fabric's appearance became purplish-pink, turned bluish-pink at pH 9.2, and eventually turned darker-blue at pH 12.

3.4 Colorimetric measurements

Figure 9 shows that at various pH solutions, CIELAB values of the fabric coloured with the synthesised dye alter as well. According to CIELAB colour space, L* constitutes lightness; coordinate a* denotes red/green hue element, and coordinate b* indicates yellow/blue hue attributes of the colour [30]. A significant change of CIELAB values has been observed in Figure 9 from pH 2.2 to pH 4.0 and from pH 8.0 to pH 12. On the other hand, a minor change of CIELAB values has been exhibited at pH values ranging from 4 to 8. In pH solution 2.2, the value of CIE L* is considerably higher, the value of CIE a* is positive, and the value of CIE b* is also positive.

For this reason, the sample became lighter, reddish, and slightly yellowish, and the surface colour of the dyed fabric appeared orange in the solution. At pH 3.0, the value of CIE L* is decreased, the value of CIE

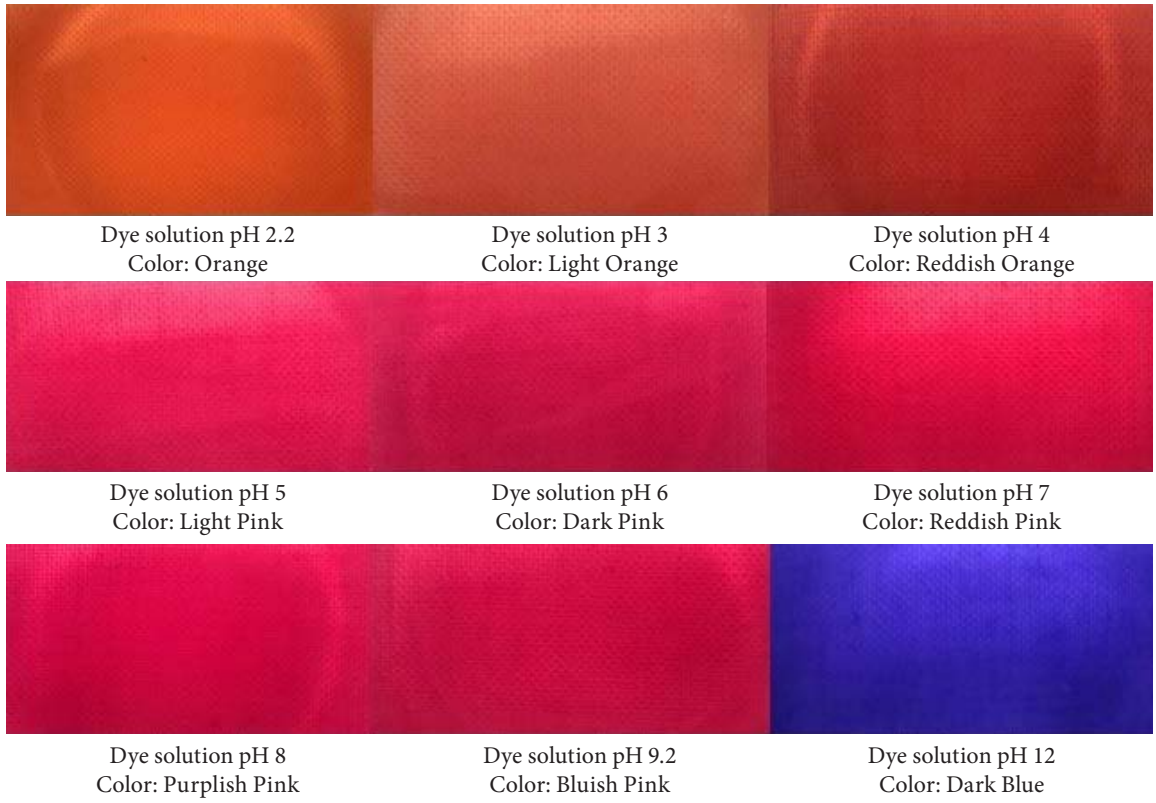

Figure 8: Colour change of cotton fabric dyed with synthesised dye in different pH solutions

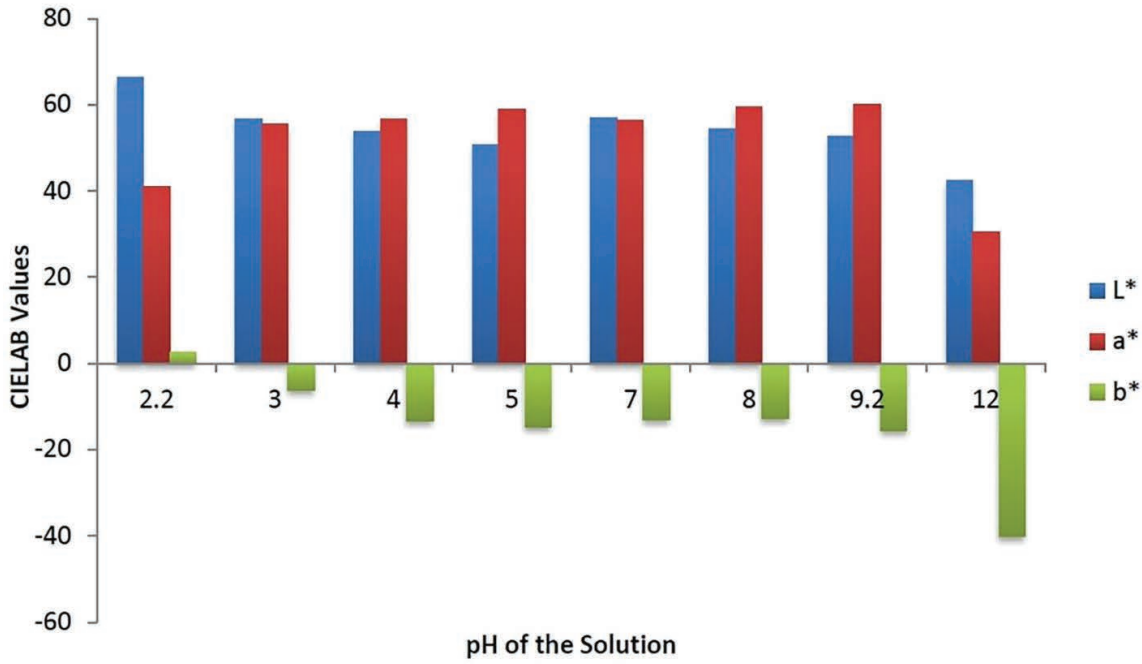

Figure 9: CIE LAB values of dyed fabric at different pH solutions

a* is increased and positive, whilst the value of CIE b* is decreased and turned to negative. The sample became darker, more reddish, and marginally blue. The decrease of CIE b* value usually brings a bluish tone to the fabric colour [31]. At pH 4, the sample became darker, slightly reddish, and bluer as the CIE L* value decreased, a* slightly raised, and b* decreased significantly. The visual appearance of the fabric turned reddish-orange in the solution at pH 4. From pH 4 to pH 8, because of the slight variation of a* and b* values, the fabric's colour appeared in marginally different shades. The fabric's colour eventually became bluish-pink at pH 9.2 due to a lower CIE b* value. Finally, in a powerful alkaline solution (pH 12), the value of CIE a* and CIE b* became the lowest, while the colour of the fabric shifted from bluish-pink to dark-blue.

The CIELAB values of dyed fabric immersed in different pH solutions are presented in Figure 9.

3.5 Wash fastness test analysis of fabric dyed with synthesised dye

The results of washing fastness of dyed cotton fabric are presented in Table 3.

The colour fastness to washing test of fabric dyed with synthesised reactive dye is assessed visually using a grey scale. The results, depicted in Table 3, showed that dyed fabric had very good (rating of 4–5) wash fastness properties. Furthermore, the dyed fabric showed slight to negotiable colour staining (rating of 4–5) on the adjacent cotton fabric. This result can be attributed to the elimination of unfixed dye molecules from the surface of the coloured cotton fabric during the wash fastness test, which then shifted to the adjacent white cotton fabric [32]. The synthesised reactive dyed fabric exhibited very good wash fastness because of the chemical fixation of dye molecules to the fibre surface through strong covalent bond formation, which ultimately resulted in the dyes' resistance to fading after washing [33]. Based on the obtained results, it can be concluded that the synthesised dye demonstrated a wash fastness rating analogous to the commercial reactive dye.

3.6 Thin-layer chromatography (TLC) analysis

The TLC analysed plate of the synthesised dye is shown in Figure 10.

Figure 10: TLC plate for the synthesised dye after the solvent almost reached the top of the plate

It is evident from Figure 10 that the TLC plate contains only one component, which appeared as a large red smear. This component is slightly spread on the TLC plate. Here, the distance travelled by the mixed solvent solution is 7.4 cm on the TLC plate. On the other hand, the distance travelled by the dye component was 4.6 cm. As a result, the R_f value of this component was 0.62. It is assumed that this component was the synthesised azo-based dichlorotirazine reactive dye. The value of R_f indicates synthesised dye is moderately polar. Based on the identification of only one component in the TLC analysis, the synthesised dye is deemed reasonably pure [23].

Table 3: Colour fastness to washing assessment of dyed cotton fabric

Change of colour	Visual assessment using grey scale					
	Colour staining of adjacent multifibre fabrics					
	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate
4/5	4/5	5	4/5	4/5	4/5	5

3.7 Response time analysis

The response time was recorded for colour changes of synthesised halochromic dye and colour changes of synthesised dyed cotton fabric in various buffer solutions. It was observed that the change in the colour of synthesised dyes occurred immediately after immersion in various pH solutions. On the other hand, it took a comparatively longer time to visualise a noticeable colour change when the dyed fabric was immersed in various buffer solutions. The response time of the cotton fabric sample dyed with synthesised halochromic reactive dye was approximately 25–30 minutes to visually notice a colour change in the fabric surface in various pH solutions. From this experiment, it is evident that, apart from the difference in halochromic behaviour, there is a substantial variation of retention time for synthesised dyes in solutions and fibrous textile matrices. The probable reason for this might be the gradual wetting properties of textile fabric, the strength of the interaction between the dyes and fibres, and the molecular structure [14, 34].

Generally, it can be assumed that this pH-sensitive reactive dye can be successfully applied to cotton fabric using a standard colouration process. However, it was observed that the diazo component used in this synthesis process produces some impurities. It is thus essential to recognise these impurities and address their effect on a dye's solubility. On the other hand, a significant difference in colour change in the solution stage and coloured fabric stage under various pH conditions was observed and may be because halochromism differs based on the constructional nature and molecular density of the fibrous substrates. Moreover, difficulties were encountered by dye molecules in accessing the fibre surface due to the slow wettability of fabric, and in the formation of a covalent bond between the dye and the hydroxyl group of cellulose, while after being applied to the fabric surface, dye molecules were immobilised as the surrounding micro-environment of the dye changed from solution to the fibrous textile matrix [14, 34]. This variation can be minimised in future work by synthesising halochromic reactive dye with a higher reactivity, more solubility, the higher affinity of dye molecules to the fabric surface, and the selection of a lower-density and more hydrophilic fabric matrix. As a result, the dye might show a comparatively similar level of colour transition both in the solution stage and the dyed fabric stage.

4 Conclusion

The fundamental aim of this research work was to synthesis an azo-based dichlorotriazine pH-sensitive reactive dye that will show a change in colour with the alteration of the pH solution. The colour change of this dye was observed in the solution and the dyed fabric stage. The synthesised reactive dye exhibited different halochromic properties in various pH environments. The dye synthesised from H-acid and 4-nitroaniline showed a colour transition from reddish-orange in pH 2.2 to red in pH 4.0, and eventually turned blue in alkaline conditions. In the dyed fabric, the colour turned orange to reddish-orange in the pH range 2.2–4.0, became light-pink at pH 5.0, turned bluish-pink in alkaline conditions (pH 9.2), and finally became deep-blue at pH 12. The halochromic dye synthesised in this research demonstrated colour transition from acidic to the alkaline environment both in the solution stage and dyed fabric state. As a result, the effectual application of halochromic reactive dye on cellulose fabric as textile pH sensors, such as those used to monitor wound healing through a change in colour on a wound dressing, might play a crucial role in the area of medical textile materials. However, some variation of colour change in the solution and dyed fabric stage was observed, and might be due to a change in the dye molecules' surrounding microenvironment after incorporation into the fibrous matrix. Overall, it can be concluded that the azo-based reactive dye synthesised in this research and applied to cotton fabric using a standard reactive dyeing process, as well as the dyed textile material itself, demonstrated decent halochromic attributes and could be used as textile pH indicators.

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Colour Memory Analysis for Selected Associative Colours
Analiza barvnega spomina za izbrane asociativne barve

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Abstract
Colours are one of the most important factors in everyday life. The exact number of existing colours is not yet fully known. Nevertheless, people are known for having poor colour memory. The ability to remember colours depends both on the characteristics of an individual and the situation in which the colour needs to be recalled. The field of colour memory (perception and memory of unusual colours) has been very poorly researched. The aim of this study was to analyse long-term colour memory for selected associative colours, comparing it with short-term colour memory. The research approach was based on observation, with observers observing for a period of time a particular colour, image, or a descriptively given reference colour. Colour was treated separately from associations in the first part, and related to associations in the second and third parts. The first part contained all the reference colours shown independently of associations, the second part contained grayscale images of brands, and the third part comprised descriptively given colours. The result analysis showed that people remember colours very poorly. Observers generally performed better in testing short-term memory. Moreover, the way the template was presented had a noticeable effect on the long-term colour memory. When the image was given in grey, the results were better. The descriptive rendering of reference colours shown did not contribute to better results. The gender of observers did not significantly affect the results.
Keywords: associative colours, colour memory, colour perception, colour difference

Izvleček
Barve predstavljajo enega izmed najpomembnejših dejavnikov v vsakdanjem življenju. Točno število obstoječih barv še ni povsem znano. Znano pa je, da imajo ljudje slab barvni spomin. Sposobnost pomnjenja barv je odvisna tako od značilnosti posameznika kot tudi od situacije, v kateri nastopi potreba po priklicu barve. Področje barvnega spomina, zaznavanje in pomnjenje nevsakdanjih barv je zelo slabo raziskano. Namen dela je bila analiza dolgotrajnega barvnega spomina za izbrane asociativne barve in primerjava s kratkotrajnim barvnim spominom. Raziskovalni pristop je temeljil na opazovanju vzorčnih predlog. Opazovalci so določen čas opazovali izbrano barvo, podobo ali opisno podano referenčno barvo. Barva je bila v prvem delu obravnavana ločeno od asociacij, v drugem in tretjem delu pa se je navezovala na asociacije. Prvi del je vseboval vse referenčne barve, prikazane neodvisno od asociacij, drugi sivinske podobe blagovnih znamk, tretji pa opisno podane barve. Rezultati so pokazali, da si ljudje zelo slabo zapomnijo barve. Opazovalci so se v splošnem bolje odrezali pri testiranju kratkoročnega spomina. Način podajanja predloge je opazno vplival na dolgoročni spomin in barvne razlike. Ko je bila predloga podana kot sivinska podoba, so bile razlike manjše, opisno podajanje referenčnih barv pa ni pripomoglo k boljšim rezultatom. Spol opazovalcev ni opazno vplival na rezultate.
Ključne besede: asociativne barve, barvni spomin, zaznavanje barv, barvna razlika

1 Introduction

Human senses form the foundation of a person and their existence. Our smell, taste, touch, hearing and sight play a key role in our understanding of the world. We use our senses to receive information from the environment. In this way, we also obtain information about various brands and companies. In consequence, the so-called "Sensory marketing", i.e. effect on customer well-being, perception and behaviour, was invented. The aspect of vision proved to be the most decisive in this field. People start explaining visual impressions of surroundings at a very early age. Most consumers thus have complete confidence in their vision. It allows them to do almost everything, from performing everyday tasks to distinguishing between different packaging and brands in the store. Visual information is extremely influential and the most important visual element turned out to be colour. Colours carry meaning and communicate information. Scientists have found that colour arrangements affect attitude as well as feelings and mood [1]. Our age and gender significantly influence which colour patterns we prefer. Fakin et al. found that in general the most popular colours are blue and green, with blue prevailing among male observers. Brown and pink turned out to be the least popular colours. The results varied throughout different age periods. One of the more noticeable changes was the popularity of black, which has grown in recent years in the younger population and has become less popular in elder age groups [2].

People update and build their archives of colour impressions on a daily basis, facing new experiences. They can name these impressions; however, they cannot avoid making mistakes when trying to recall them from their long-term memory. A comparison of a colour in the current situation with the one from the past happens completely automatically, naturally, yet the choice and the results vary depending on the circumstances and colour shades [3]. The ways of testing colour memory are very different. Perez-Carpinell stated [4] that colour memory is successive colour matching after a certain time has elapsed from the observation. Comparing the colour from our long-term memory with the present is much more important as it may seem at first glance. People choose fresh fruits and vegetables based on their previous experience, which means freshness, ripeness. They usually select and buy clothes according to their colour preferences and they pick the colour that matches the rest of their outfit [5].

A simultaneous comparison of samples with the reference colour is usually very accurate. The results of the research confirmed as many as 96% correct results. In the case of the remaining 4%, the colour difference was minimal [6]. A successive comparison occurs when some time elapses between the observation of a given reference colour and the sample. In this case, the colour memory is used, which is more common in everyday life [4]. Research has also confirmed that the more we increase the pause time, the greater the colour differences; however, only to a certain extent. If increased over 15 min, no major differences are observed [7–10].

Bodrogi and Tarczali [11] studied how colour memory is affected by the surroundings of a colour pattern, when it is observed within a certain image or context. Prototype paints or associative colours, e.g. the colour of the sky, plants, and skin, were observed as a simple colour pattern shown in a photorealistic image. The results showed that the association could be influenced by the added image despite the longer time period having passed since remembering the colour stored in long-term memory.

The aim of our study was hence to examine how the method of recall from memory affects our long-term memory. To examine this, we used in addition to independent colour patterns two options, i.e. grayscale images of brands and a description of associative colours. A comparison of short-term and long-term memory was performed on the basis of calculated colour differences.

2 Experimental

The experimental part was based on an observation experiment, which was divided into three parts. In the first part, observers were exposed to a single colour for 5 s, then after a 10 s pause, they used a circular template to select the colour they thought was the reference. The set of colours used in the first part was then repeated in the second and third part. The first part thus contained 16 colours, and the second and third contained 8 colours each. The second part contained grey images of certain brands, and the third part included descriptions of associative well-known colours. In addition to short-term memory, we also tested long-term memory. In the first part of the study, colours were considered independently of

associations, and in the second and third part, they were considered in conjunction with corresponding associations.

2.1 Preparation of reference colours and patterns

Reference colours were divided into two groups, each containing 8 colours. The first group (cf. Table 1) contained associative colours that are tied to everyday experiences, i.e. cinnamon brown, grass green, sky blue, cyan, lemon yellow, colour of an orange, purple red and magenta. The second group (cf. Table 2) consisted of associative colours related to brands and companies, i.e. Starbucks green, blue colour of the European Union, Facebook blue, Milka purple, yellow colour of the Post office Slovenia, Mueller orange, red colour from the University of Ljubljana and red-pink colour of the Mercator store.

We checked the representative colours of companies online and in collections. Those related to descriptive naming were selected according to the colour values that were reported most often. Colour values were presented in the *CIELAB* colour space using $L^*a^*b^*$ coordinates [12].

All reference colours and associated patterns were prepared with Photoshop. The entire template was made in InDesign to ease the reading of the results. The method of selection and the conditions taken into account are described below.

Selection of samples according to each reference colour

For each reference colour, we prepared 8 different visually similar colour samples, which were selected according to three basic colour properties, i.e. hue, lightness and saturation (cf. Figure 1). Samples were

Table 1: Reference colours with CIE $L^*a^*b^*$ coordinates; Group 1: colours of well-known objects

Reference colour	Sample	L^*	a^*	b^*
1-I		56	38	56
1-II		48	–23	25
1-III		79	–18	–22
1-IV		91	–51	–15
1-V		95	–10	76
1-VI		68	45	74
1-VII		56	76	69
1-VIII		60	93	–61

Table 2: Reference colours with CIE $L^*a^*b^*$ coordinates; Group 2: colours of brands and logos

Reference colour	Sample	L^*	a^*	b^*
2-I		37	–36	19
2-II		15	46	–77
2-III		38	4	–39
2-IV		39	25	–43
2-V		84	9	83
2-VI		61	52	62
2-VII		48	66	53
2-VIII		48	72	26

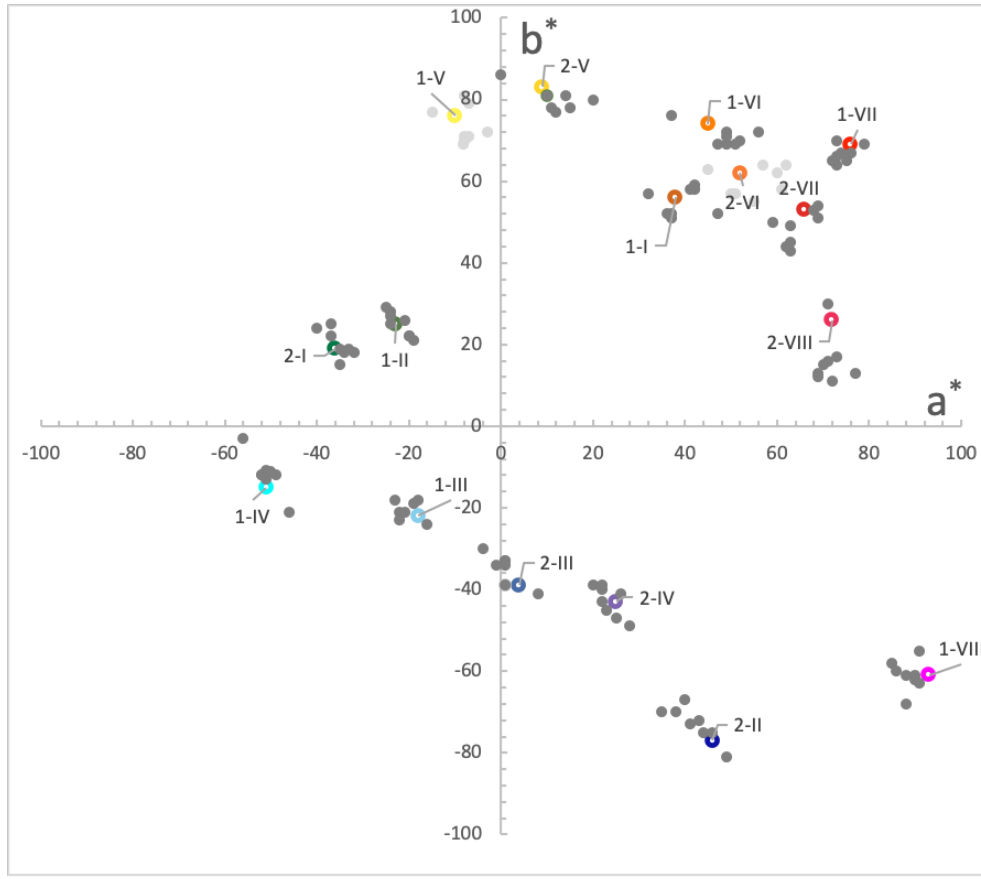


*Figure 1: Reference colours with appropriate samples in a*b* plane of CIELAB colour space*

obtained by changing the *CIELAB* hue difference, ΔH^*_{ab}, by 2 units, *CIELAB* lightness difference, ΔL^*, by 3 units, and saturation, i.e. *CIELAB* chroma difference, ΔC^*_{ab}, by 3 units. An exception was the blue colour of the European Union, where the samples did not differ enough from each other for the observer to be able to distinguish among them; therefore, we changed them by 5 units (–5, –10 and –15).

2.2 Test preparation

Test group

The test group consisted of 12 observers, 8 female and 4 male. The age range was 15–30 years, since people are most sensitive to perception in this period [11, 13]. The oldest observer was 24 years old and the youngest was 16 years old, for at younger observers deviations could occur [8]. In accordance with recommendations [9], all participants previously performed the Farnsworth-Munsell hue colour vision test to demonstrate their ability to distinguish colours and assure their normal colour vision. Observers had different educations in different fields of study. Some also had poorer eyesight and used glasses; however, this did not affect the test results.

Observation conditions

The conditions of observation were the same for all observers, ensuring comparable results and excluding the influence of possible external factors. A 25-inch Dell U2518D monitor with the resolution of 1920 × 1080 and brightness of 350 cd/m^2 was used. Brightness was set to maximum value. The testing was performed in a dark room, the only light source being the screen.

The observer was positioned 50 cm away from the screen, sitting at a 90° angle to the screen. Before each test, we checked the screen brightness and the display resolution of the screen image.

Presentation of colour templates

For each reference colour, four different templates were prepared (cf. Figure 2). The first colour template contained only the reference colour shown in the shape of a square measuring 6 × 6 cm. The other two templates contained a reference colour and 8 associated samples. The templates differed from each other

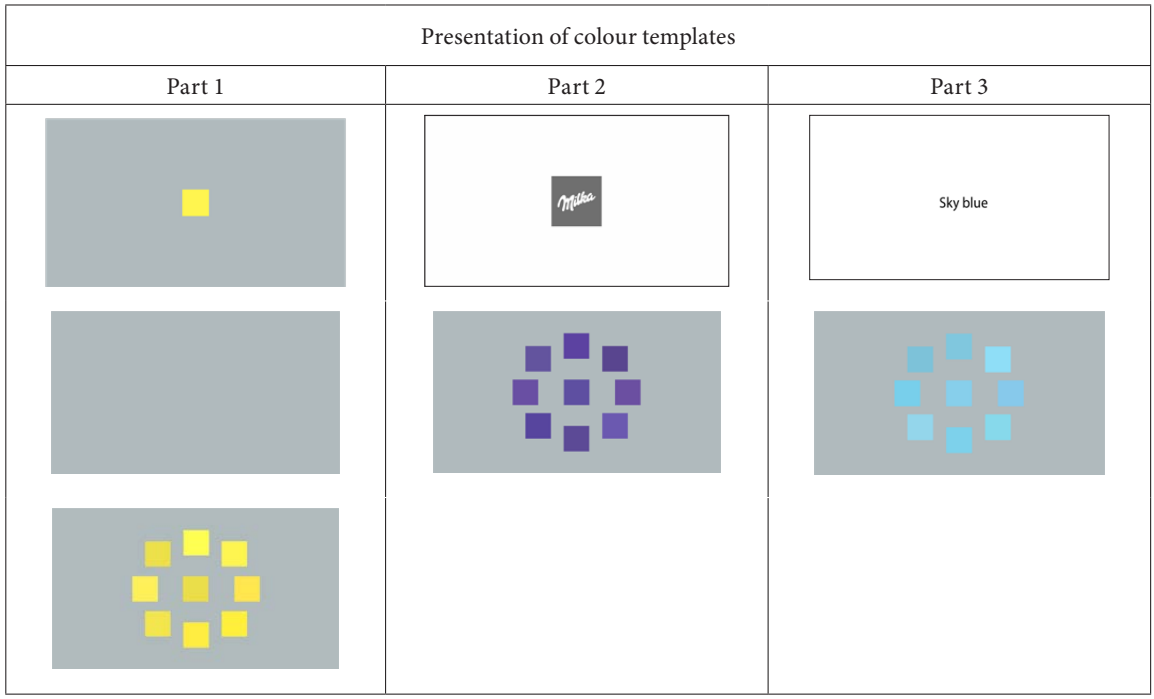


Figure 2: Presentation of colour templates when testing colour memory

in the arrangement of colour patterns. Each sample was shown in the form of a 6 × 6 cm square as well. The squares were arranged in a circle in the middle of the template. To ease the observing and reduce eye fatigue, the background colour was neutral grey ($L^* = 75$, $a^* = -3$, $b^* = -2$). The third template depended on the group the colour was from. In Group 1, i.e. tied to colour names, the template only contained a description of the colour on white background. The typography used was an 87-point Myriad Pro. In Group 2, i.e. colour tied to the brand, the image of the brand was shown in a 6 × 6 cm square in grey tones on white background. A neutral grey background was displayed for 10 s between each reference colour template and the sample template (cf. Figure 2).

2.3 Performance of testing

We first explained the course of the research in detail to each observer to have time to adjust to a dark space. The first part of the study contained all 16 reference colours from both the first and the second group, the observers not being aware of this. A template with a reference colour was displayed for 5 s, which was followed by a 10-second pause with a neutral grey background to calm the eyes and prevent the glow of colours. Studies [14] have confirmed that memorising is best in the first 5 s, prolonging the time not having any major effect on the results. The observer then selected a sample for which they considered it is the same as the reference. The time for sample selection was not limited, since this has not been shown as necessary in previous studies [14, 15]. A new template with a reference colour followed. In the second part, the observer observed grey images of well-known companies and brands. The attachment was displayed for 5 s, then they chose the colour sample for which they thought it belonged to the company. At this stage, we checked long-term memory bound to associative colours.

In the third part of the research, associative colours were given descriptively. The same as in the previous parts, the template was shown for 5 s. Based on the experience, the observer selected a colour sample that they associated with the description.

2.4 Evaluation of colour differences

The reference colours and the selected colour samples were defined by the coordinates of the *CIELAB* colour space and the colour differences, ΔE^*_{ab}, were calculated using the basic *CIELAB* equation [12]. Moreover, the contributions of *CIELAB* lightness difference, ΔL^*, saturation, i.e. *CIELAB* chroma difference, ΔC^*_{ab}, and *CIELAB* hue difference, ΔH^*_{ab}, were calculated, describing the differences between the observed reference colour and memorised colour represented by the selected sample [16].

3 Results with discussion

3.1 Overview of colour differences

In the first part of the study, where short-term colour memory was tested, male observers (ΔE^*_{ab} = 5.09) performed slightly better than female (ΔE^*_{ab} = 5.26). *CIELAB* lightness differences were minimal (ΔL^* = 0.04), similarly observed in previous studies [17]. The differences in saturation were also small (ΔC^*_{ab} = 0.29). According to the results of our study, hue was remembered the least accurately (ΔH^*_{ab} = 4.95), which contradicts with the findings of some other studies [6]. In this case, male observers performed better (ΔH^*_{ab} = 4.69) than female (ΔH^*_{ab} = 5.21) (cf. Table 3).

In the second part of the study, which was based on brand recognition, female observers (ΔE^*_{ab} = 4.99) performed better than male (ΔE^*_{ab} = 5.21), which might be due to women being more often in contact with brands and companies. Again, the *CIELAB* lightness differences were very small (ΔL^*= 0.07), the average difference in saturation being slightly larger (ΔC^*_{ab} = 1.40). The largest difference was observed as *CIELAB* hue difference (ΔH^*_{ab} = 5.00), where larger deviations were detected by male observers (ΔH^*_{ab} = 5.21) compared to females (ΔH^*_{ab} = 4.79).

In the last part, related to the conceptual representation of associative colours, the average colour difference was the highest (ΔE^*_{ab} = 5.33), which can be attributed to poor colour memory, especially unreliable long-term memory. The *CIELAB* lightness difference for selected samples was approximately one unit (ΔL^*= 1.09) and no major deviations in saturation were observed (ΔC^*_{ab} = 0.86). The largest contribution to the *CIELAB* colour difference was detected as the *CIELAB* hue difference (ΔH^*_{ab} = 5.13). The latter is unusual and in contradiction to some previous research [6], as it would be expected that this property is remembered most accurately as basic colour information. *CIELAB* lightness differences are expected to be small, although most studies show that observers remember light reference patterns as even lighter and dark as darker [7, 13] (cf. Table 3).

3.2 Comparison of long-term and short-term memory

Reference colours Group 1: well-known objects

The first group contained associative reference colours that relate to familiar concepts and objects. The results (cf. Figure 3) showed that the average colour difference for Group 1 of the reference colours was greater in Part 3 of the study (ΔE^*_{ab} = 5.27) than in Part 1 (ΔE^*_{ab} = 4.40). The first part was based on short-term memory and the third part on long-term memory. Observers had to recall only what they thought was most appropriate colour and then select a sample. Given that all observers successfully passed the colour vision test, the reason for errors was primarily their poor long-term memory for colours. The total value of the colour difference was mostly due to the *CIELAB* hue difference, which was also larger in Part 3 (ΔH^*_{ab} = 4.16) than in Part 1 (ΔH^*_{ab} = 3.70). There were no major lightness differences (Part 1: ΔL^*= 1.04 and Part 3: ΔL^*= 1.11) nor chroma differences (Part 1: ΔC^*_{ab} = 1.83 and Part 3: ΔC^*_{ab} = 1.47). On average, observers chose darker and less saturated samples. In general, we can say that the differences are greater when dealing with long-term colour memory. For most reference colours, a larger colour difference was found in Part 3 and a smaller one in Part 1.

The results showed that the best recognised reference colour was in Part 1 of the study colour 1-IV (cyan) with the smallest overall colour difference (ΔE^*_{ab} = 1.89). The reason can be attributed to the uniqueness and unnaturalness of the colour. A much larger colour difference was observed in Part 3 of the study (ΔE^*_{ab} = 6.43), when observers had to recall the

Table 3: Average colour differences in Part 1 (short-term memory), Part 2 (long-term memory using grayscale image) and Part 3 (long-term memory using description of colour)

Part	Part 1			Part 2			Part 3		
Gender	Female	Male	All	Female	Male	All	Female	Male	All
$\lvert\Delta H^*_{ab}\rvert$	5.21	4.69	4.95	4.79	5.21	5.00	5.05	5.21	5.13
$\lvert\Delta C^*_{ab}\rvert$	0.62	1.96	0.29	1.33	1.48	1.40	0.86	0.85	0.86
$\lvert\Delta L^*\rvert$	0.34	0.27	0.04	0.43	0.28	0.07	0.72	1.47	1.09
$\lvert\Delta E^*_{ab}\rvert$	5.26	5.09	5.18	4.99	5.42	5.21	5.17	5.48	5.33

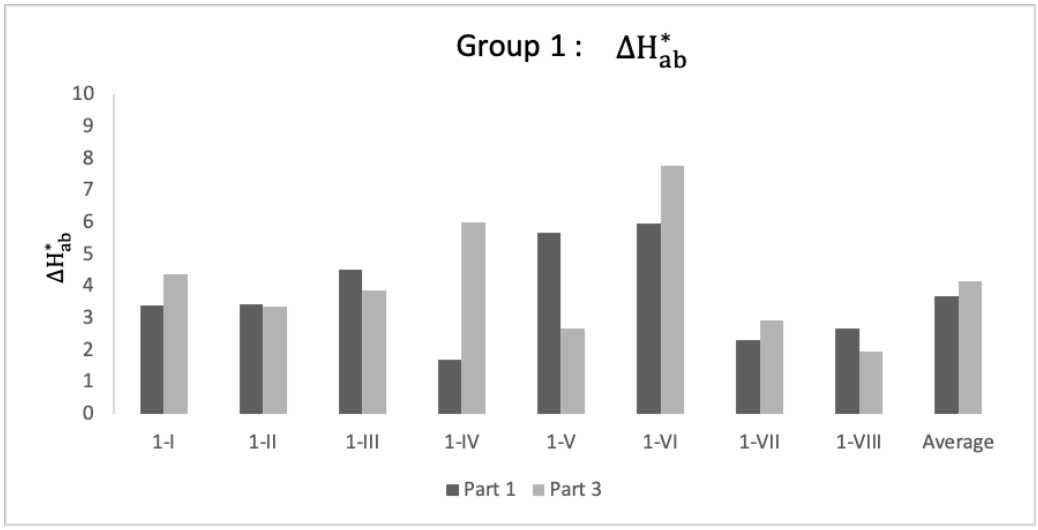
same colour from memory and select the correct pattern. Let us mention that most of the observers were full-time students in the field of graphic arts, this colour hence being well known to them. Similarly, it is worth mentioning the reference colour 1-VIII (magenta), which was also well recognised by the observers, especially in Part 3 of the study (ΔE^*_{ab} = 2.61). The worst recognised reference colours were 1-V (lemon yellow) and 1-VI (orange fruit). In both cases, the average colour differences were high, which can be attributed to the fact that both yellow and orange have a smaller number of light levels and the differences increase rapidly. We attribute the large discrepancies to our perceptions of the colour of an orange and our experience of it. A similar study was performed using a monochromatic light source that also displayed a lemon yellow colour. Otherwise, this colour is supposed to have the highest accuracy, with the wavelength peak at 570 nm (in addition to blue with the peak at 494 nm). The observers recognised it best and the results had the smallest deviations from the reference colour in a given case. Improvement followed by using the association with a lemon [14].

The biggest contribution to the total *CIELAB* colour difference was due to the *CIELAB* hue difference which in some cases almost equalled the total colour difference. All reference colours that achieved a larger total *CIELAB* colour difference in Part 3 than in Part 1 of the study also exhibited a larger *CIELAB* hue difference in Part 3 than in Part 1: 1-I (cinnamon brown), 1-II (grass green), 1-IV (cyan), 1-VI (orange fruit) and 1-VII (purple-red). Due to the predominant influence of the *CIELAB* hue difference on the total colour difference, the reverse also applies to all other reference colours.

The deviations in *CIELAB* lightness were relatively small, with the exception of the reference colours 1-I (cinnamon brown, Part 3: ΔL^*= –2.42), 1-III (sky blue, Part 1: ΔL^*= 2.83 and Part 3: ΔL^*= 2.42), 1-VI (orange fruit, Part 1: ΔL^*= –3.58) and the reference colour 1-VII (purple red, Part 3: ΔL^*= –4.67). Even when there was a larger deviation, observers chose darker samples than the reference. An exception was found only for the reference colour 1-III (sky blue), for which lighter samples were chosen.

We also detected similarly small differences in saturation when recalling colours from memory. Observers selected less saturated samples in most cases. Major deviations were only in the case of the reference colours 1-I (cinnamon brown, Part 3: ΔC^*_{ab} = 3.79), 1-VII (purple red, Part 1: ΔC^*_{ab} = –2.84 and Part 3: ΔC^*_{ab} = –3.98) and the reference colour 1-VIII (magenta, Part 1: ΔC^*_{ab} = –2.84).

The comparison of Parts 1 and 3 of the research agrees with our assumptions that the differences will be greater in Part 3, which is tied to long-term memory, and this is also in agreement with previous investigations [8, 9]. Regardless of the fact that the observers had the reference colours descriptively given, this did not affect their final decision. Each one of us has a different idea of objects; therefore, we choose different colour patterns depending on our memory. The evocation of associations by means of a verbal description of colour did thus not affect the improvement of long-term memory. The only exception may be the reference colour 1-VIII (magenta), which achieved noticeably better results when given descriptively. This colour is well known by its name and the descriptive rendering in this case led to mi-



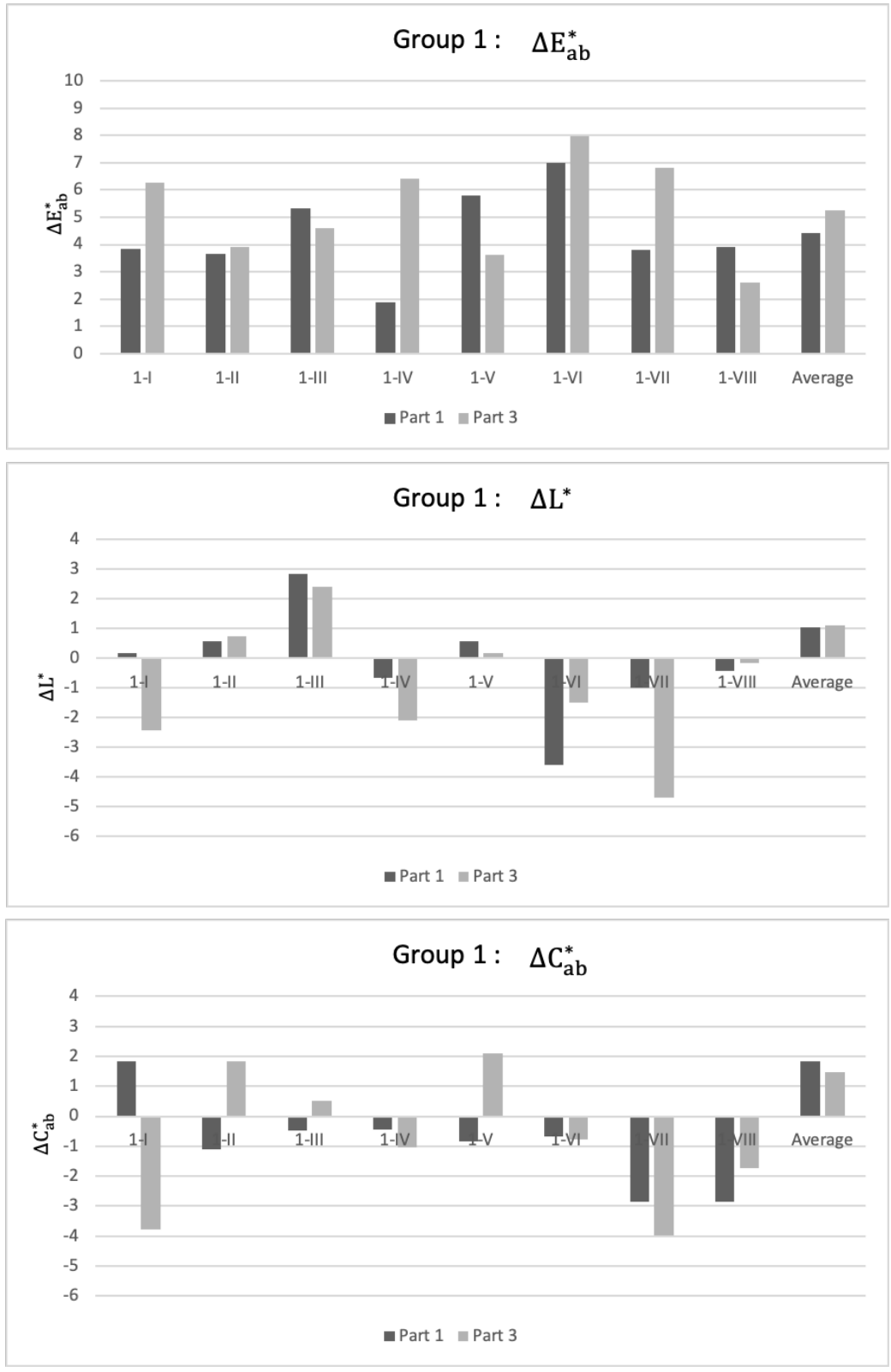


Figure 3: Comparison of Part 1 (short-term memory) and Part 3 (long-term memory using description of colour) for samples 1-I–1-VIII: CIELAB colour difference (ΔE^*_{ab}), CIELAB hue difference (ΔH^*_{ab}), CIELAB chroma difference (C^*_{ab}) and CIELAB lightness difference (ΔL^*)

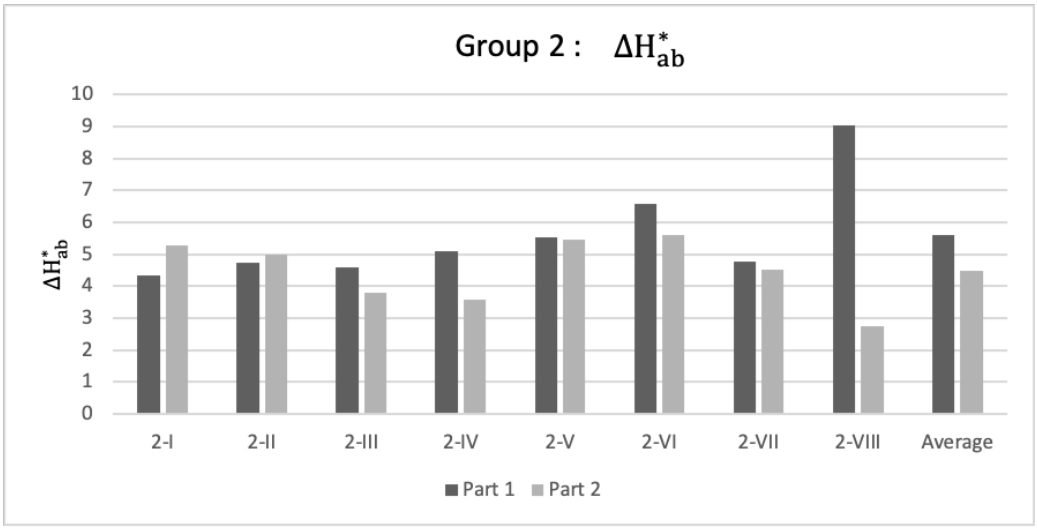
nor colour differences. The explanation for better recognition could also lie within the Weber's law [18], as its initial stimulus intensity is higher due to its chromaticity, grey background and dark room.

Reference colours Group 2: brand colours

The second group contained associative reference colours that relate to companies and brands. The results are shown in Figure 4. The average colour difference in Part 1 of the study was $\Delta E^*_{ab} = 6.01$ and in Part 2 $\Delta E^*_{ab} = 5.13$. Contrary to our expectations, the results were better in Part 2, when observers selected samples based on long-term memory. The reason can be found in the fact that most observers are often in contact with the colours of the brands that were presented as a reference. Whenever there is a connection between a colour and an object or an image from our memory, there are differences in selected patterns and thus in research results. An improvement and a smaller deviation of the overall colour difference was observed compared to the situation where there were no associations [15, 19].

The results for the reference colours 2-III (Facebook blue), 2-IV (Milka purple), 2-VI (Mueller store orange) and 2-VIII (red-pink colour of the Mercator store) were consistent with the findings of a smaller colour difference in Part 2. The reference colour 2-VIII achieved the largest colour difference within Part 1 ($\Delta E^*_{ab} = 9.42$) and the smallest colour difference within Part 2 ($\Delta E^*_{ab} = 2.75$) as it was best recognised. All observers recognised this brand very successfully. The reference colour 2-VI (Mueller store orange) was less recognisable (Part 1: $\Delta E^*_{ab} = 6.68$ and Part 2: $\Delta E^*_{ab} = 5.62$), perhaps due to less frequent encounters with it, or just a human tendency to remember bright colours less well. In the case of the reference colour 2-III (Facebook blue), the differences (Part 1: $\Delta E^*_{ab} = 5.87$ and Part 2: $\Delta E^*_{ab} = 5.00$) occurred most likely due to different screen renderings of the application of the mentioned social network and the previously changed representative colour of the application. The reference colour 2-IV (Milka purple) was very well recognised by most observers (Part 1: $\Delta E^*_{ab} = 5.35$ and Part 2: $\Delta E^*_{ab} = 3.86$). In fact, they had bigger problems in Part 1, when they had to imprint the colour in their memory and recognise it after 10 seconds.

Interestingly, the reference colour 2-VII (red colour of the University of Ljubljana, Part 1: $\Delta E^*_{ab} = 5.53$ and Part 2: $\Delta E^*_{ab} = 5.69$) achieved very similar colour differences in both parts of the research. Due to the fact that all observers are in frequent contact with this colour, such results differ from expectations in the case of long-term memory and can be explained by a variety of representations, as the problems are mainly a consequence of inconsistent rendering and rendering of colours; the overall graphic image of the University of Ljubljana uses a darker colour than the website. The reason for the deviation of the reference colour 2-I (Starbucks green) is probably that its recognition depends on the frequency of encountering the brand. The observers who are not very familiar with it consequently did not recognise it well in Part 2 of the study. The reference colour 2-II (blue colour of the European Union) made greater differences (Part 1: $\Delta E^*_{ab} = 4.95$ and Part 2: $\Delta E^*_{ab} = 6.19$), most likely due to the inconsistency in its representations (flags, screens, application, TV etc.). Each participant has thus a completely different idea of this colour.



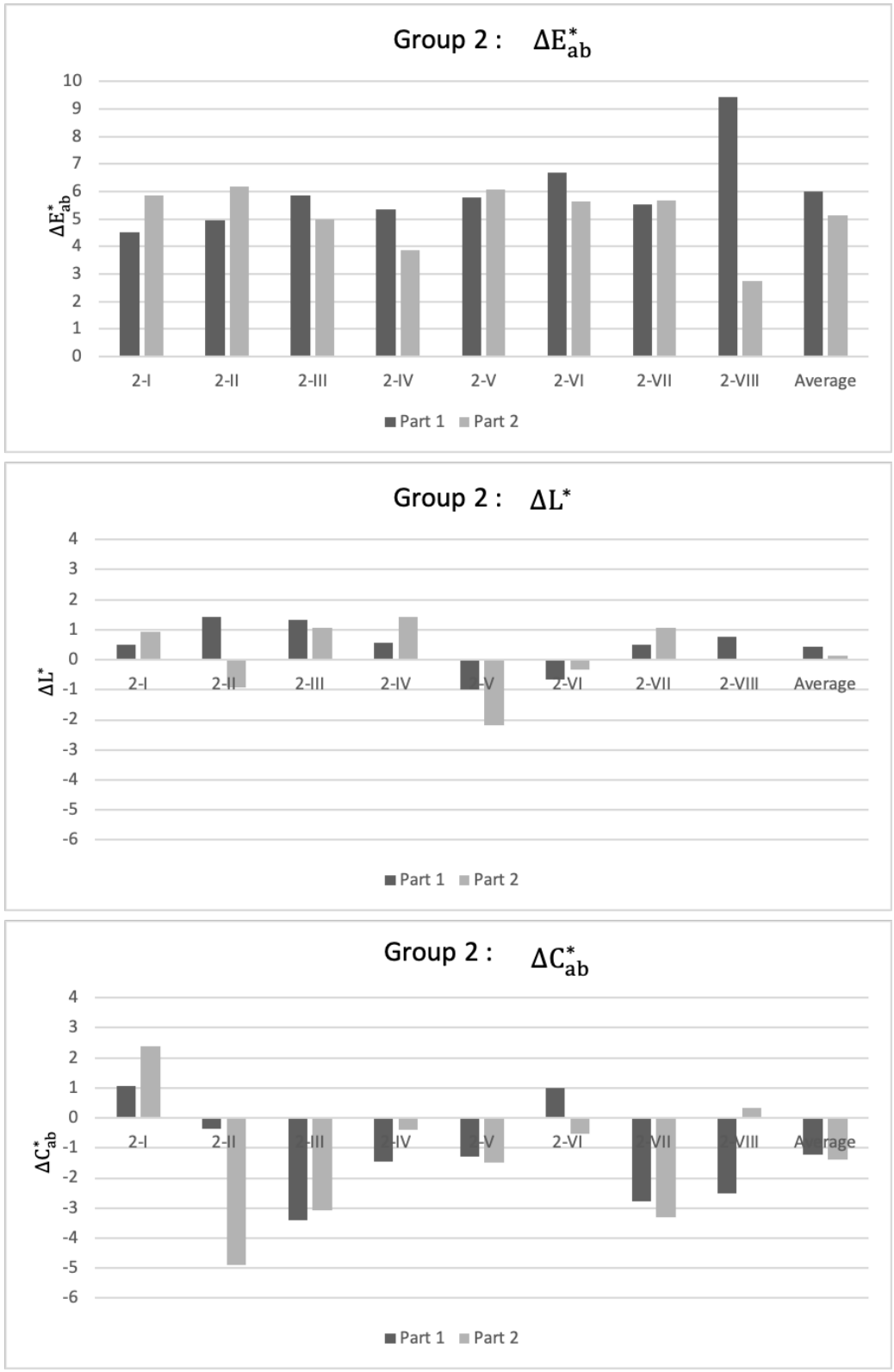


Figure 4: Comparison of Part 1 (short-term memory) and Part 2 (long-term memory using grayscale image) for samples 2-I–2-VIII: CIELAB colour difference (ΔE^*_{ab}), CIELAB hue difference (ΔH^*_{ab}), CIELAB chroma difference (C^*_{ab}) and CIELAB lightness difference (ΔL^*)

The average *CIELAB* lightness differences were very small in both Part 1 and 2 of the study (Part 1: ΔL^*= 0.43 and Part 2: ΔL^*= 0.14). Generally, observers chose lighter samples than the reference colour.
The average *CIELAB* chroma differences were slightly larger (Part 1: ΔC^*_{ab} = –1.21 and Part 2: ΔC^*_{ab} = –1.37). Observers mostly chose less saturated samples.
The colour differences were predominantly displayed as the *CIELAB* hue difference (Part 1: ΔH^*_{ab} = 5.59 and Part 2: ΔH^*_{ab} = 4.49), which again had the greatest impact on the total colour difference. Consistent with the total *CIELAB* colour difference, the *CIELAB* hue difference was greater in Part 1 than in Part 2 for the majority of Group 2 reference colours.
The comparison of Parts 1 and 2 of the research does not match our assumptions that the differences will be greater in Part 2, which depended on long-term memory. The differences were smaller in Part 2, where observers selected samples according to the grey image of the brand. Evidently, the way the suggestions were made was crucial for minor colour differences and had an impact on better long-term memory results. Similar results were found in a research when observers used a black and white photography of a reference coloured object [20]. According to the results, observers performed better in Part 2 of the study when observing grayscale brand suggestions, with some exceptions that were either not well known among observers or differed in the ways in which they were depicted and the applications they encountered: 2-I (Starbucks green), 2-II (blue colour of the European Union), 2-V (yellow colour of the Post office Slovenia) and 2-VII (red colour of the University of Ljubljana). According to the Weber-Fechner law, the perceived magnitude of a stimulus, in this case colour, is proportional to the logarithm of the physical stimulus intensity [21]. Consequently, such results could reflect the inability of the human visual system to distinguish relatively small colour differences in case of highly saturated colours.

4 Conclusion

The result analysis confirmed that people have a deficient memory for colours. Observers performed much worse in the part of the study that was tied to long-term memory. We can therefore confirm that our long-term memory is not as accurate as short-term. Although an unreliable colour memory can lead to unpleasant surprises when selecting a certain hue, e.g. when buying clothes, this can be improved by offering suitable support or association. The results showed that the way colour suggestions are made has a significant impact on colour differences when testing colour memory. When the suggestions were given only with the help of verbal descriptions of reference colours, the results were worse, consequently confirming our hypothesis that deviations are greater with long-term memory. In the case of grayscale brand proposals, however, observers achieved better results. Here, the association with the help of a grayscale template had a strong impact on improving long-term memory. The results showed that our memory for lightness is relatively accurate. In general, the colours in our memory are slightly more saturated than they really are. The largest share of the total colour difference was exhibited as the hue difference, which is in contradiction to some previous research. Female observers remembered the colours slightly better than male, the differences between the two genders not being substantial.

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