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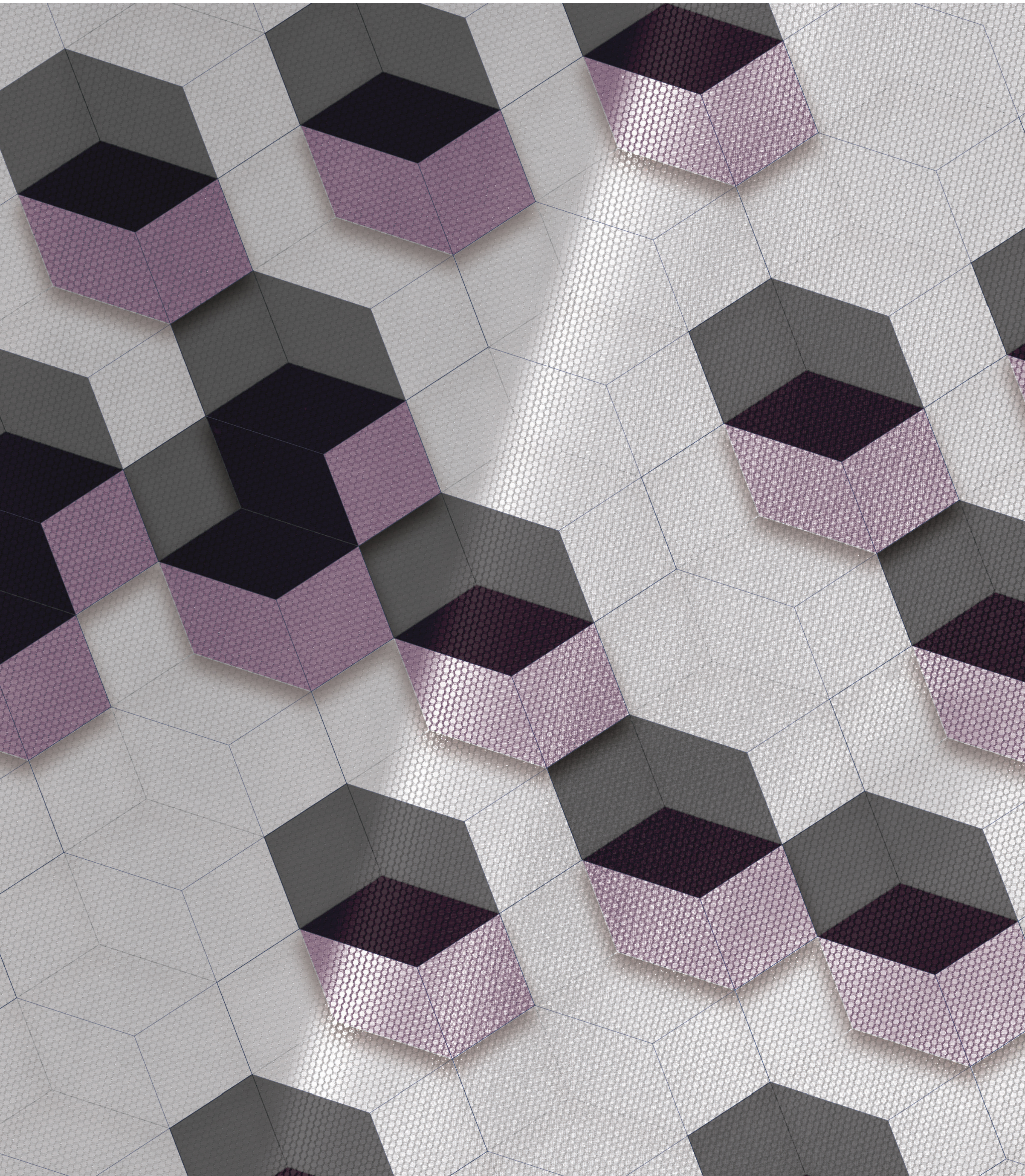

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Effect of Service Quality on Service Value and Customer Retention for Clothing Store Brands in China
Vpliv kakovosti storitev na njihovo vrednost in na to, koliko so kupci zvesti trgovskim blagovnim znamkam oblačil na Kitajskem

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Abstract
An increasing number of new Chinese clothing store brands are selling and offering similar products and services and consequently clothing store brand providers must compete to survive in this industry. They need to focus on customers' special needs and preferences to maintain and retain a long-term relationship. The objective of this study is thus to examine the relationship between service quality and customer retention for clothing store brands in China, and the mediated effect of service value in the relationship between service quality and customer retention for clothing store brands in China. A quantitative research for data collection was implemented. As many as 385 questionnaires were collected by the professor, PhD students, MSc students and BSc students of different nationalities in China. The data was analysed using SmartPLS and SPSS software. Customer perception of the quality of a service product in all sizes has a beneficial effect on customer retention. Service value affects customer retention positively. Practical implications for the target market of the clothing sector should be focused on young individuals aged 36 to 45 years, holding a Master's degree and earn more than €1,000/month. Findings indicated significant and direct relationships between service quality, service value and customer retention. It was also found that service value has a full mediated effect. This study will be of interest to the clothing store brands in understanding how service quality is essential for maintaining a long-term relationship with customers.
Keywords: service quality, service value, customer retention, clothing store brands, China

Izvleček
Na Kitajskem je v prodaji oblačil s trgovskimi znamkami veliko novih subjektov, ki tržijo podobne izdelke in storitve. Ponudniki oblačil uveljavljenih znamk morajo tekmovati med seboj za preživetje, saj je konkurenca zelo huda. Da bi ohranili lojalnost svojih kupcev na dolgi rok, so prisiljeni upoštevati posebne potrebe in želje svojih strank. Cilj te študije je torej proučiti razmerje med kakovostjo storitev pri ohranjanju zvestobe trgovskim znamkam oblačil na Kitajskem in posrednim učinkom vrednosti storitve na razmerje med kakovostjo storitev in zvestobo trgovcem z oblačili na Kitajskem. Izvedena je bila kvantitativna raziskava z zbiranjem podatkov. Anketiranih je bilo 385 profesorjev, doktorskih študentov, magistrov in študentov univerzitetnih študijskih programov na Kitajskem. Podatki so bili analizirani s programsko opremo SmartPLS in SPSS. Dojemanje storitev kot kakovostnih v vseh vidikih ugodno vpliva na zvestobo

kupcev. Vrednost storitve pozitivno vpliva na zvestobo strank. Iz tega sledi, da bi se na ciljanih trgih oblačil morali osredotočiti na mlade, stare od 36 do 45 let, ki imajo magisterij in na mesec zaslužijo več kot tisoč evrov. Pokazalo se je, da obstajajo pomembne in neposredne povezave med kakovostjo storitve, vrednostjo storitve in zvestobo strank. Ugotovljeno je bilo tudi, da ima vrednost storitve popoln mediacijski učinek. Ta študija je zanimiva za trgovine z blagovnimi znamkami oblačil, da bi laže razumeli, kako pomembna je kakovost storitev za ohranjanje dolgoročnih odnosov s strankami.
Ključne besede: kakovost storitev, vrednost storitve, zvestoba kupcev, trgovska znamka oblačil, Kitajska

1 Introduction

The clothing store brands nowadays not only meet customers' primary needs, such as food and shelter, they also provide much more, for example they can cover other requirements, such as personalised services, etc. As many organisations and sectors are evolving, the Chinese clothing sector has become one of the most critical drivers of Chinese economic growth and development. China was the leading global economy in 2019 [1].

In a competitive clothing sector, clothing store brands need to discover methods to improve their services through various and comprehensive programs that are different from their competition. In order to achieve this goal, clothing store brands managers need to understand and find out the needs and anticipations of their customers. Then they need to alter their product service offerings based on the needs and expectations of their customers to properly satisfy their needs [2]. In order to enhance customer retention in the clothing sector, clothing store brands need to understand that clients may be influenced by service characteristics. Inability to provide vital attention to the quality of services (tangibles, assurance, reliability, responsiveness and empathy) can lead to a negative evaluation of the clothing brands by customers, which can ruin the brand's opportunity of having more customers. Recent research has shown that consumer retention in the clothing sector can be affected by numerous marketing factors, such as promotion, price, place, product, people, process, physical evidence and after-sales service. These factors are considered essential [2] in order to achieve consumer loyalty and satisfaction.

According to one source [3], the combination and mixing of all parameters affecting the retention of customers lead to the quality of the service. Knowing the impact of service quality on service value and customer retention is therefore important for clothing store brands. Since the retention of a customer is affected by the quality of the service, the marketing mix has become a major issue for all companies [3]. This study seeks to investigate the impact of service quality on service value and customer retention for clothing store brands in China.

2 Materials and methods

2.1 Customer retention

Customer retention means an undertaking's ability to continue business with a specific customer or constantly adapt to their needs. Retention can also be described as love, identification, engagement, confidence, and clients' readiness to recommend and repurchase intentions; the first four expressions are emotional-cognitive retention constructs and the last two behavioural intents [4]. According to Oliver and Varki [5] customer retention is a strong commitment to continuously repurchase or repatriate a preferred product or service in the future, despite situational factors and marketing attempts that may trigger the switching of behaviour. Later, Ranaweera and Prabhu [6] define customer retention as the customer's propensity to remain with its current service provider. Many organisations today have trouble attracting new customers so their marketing department is restructured and managers are appointed to pay attention to their current customers [7]. Customer retention is very important for the clothing industry in order to ensure long-term sustainability and growth and it is therefore their duty to be able to meet all of the customers' needs. They must be aware of the current situation whenever crises occur and be prepared to react rapidly. In addition, the management of the clothing store brands must guarantee that individual customers are as satisfied as possible. If the leadership of clothing store brands is serious about contributing to the success of their clients, then retention is a key element. In reality, the minimal expectation in terms of retention should be that clients will simply return to the clothing store brands withing the first year [8].

2.2 Service value

Creation of value derives from services [9, 10]. Prior to choosing a service, customers are smart enough to consider and determine any advantages these services may have. Such a scenario has encouraged the company to provide benefits and services from the customer's point of view with a useful service for competitive benefits, profitable growth and business development.

The concept of 'value development' becomes an important measure in consumer behaviour, such as customer retention and behavioural intentions that are in line with increasing recognition of service value [11, 12]. Furthermore, building customer relationships that will lead to development and profitability [13] as a key aspect in the business strategy [9] is crucial for the organisation's continued growth and development. This implies that providing greater value to clients will allow organisations to: (1) reinforce profit and maintain a competitive advantage by incorporating significant strategic directions [11, 14]; (2) simplify resource allocation by scheduling managers linked to service design and the delivery stage [15]; and (3) improve service encounter systems that drive customers to achieve beneficial results [16].

Despite a wide-ranging attention and previous research, several problems connected with the core concern of service value remain uncertain. Wu and Ledden to studies by some authors [12, 17], unresolved problems relating to the importance of the service cover (1) the definition and idea are lacking in compromise. Consequently, the structure of service value has always been misapplied in social sciences and marketing research of goods in particular [18]; (2) The findings from the empirical measure are inconsistent and the topic has been criticised for lack of agreement between academics on definitions and ideas; and (3) the connection between service value and other constructs, such as service quality and customer retention has been controversially discussed.

As value-related problem discussions are yet to come to an agreement on generalisation, ongoing research of this structure is required to narrow the knowledge gap as this research attempts to explore the value of service including links to other constructs. In the Thamrin [19] study, they believe that the value of the service is situational and context-dependent. Due to its nature, the value judgment is a function of evolving norms. These norms are likely to differ depending on the environments, location, culture and the moment the value assessment was conducted.

2.2.1 Relationship between service value and customer retention

Service value and customer retention are key elements of academic and business marketing because these factors are closely connected to market share, marketing relationships and purchase intention, as well as behaviour [20]. In the literature on marketing services the connection between service value and customer retention has thus been seriously deliberated.

As a background and result, there were two kinds of customer retention roles to service value. According to Oliver's model [21], the first form of customer retention may lead from results of performance, such as product efficiency, quality of service or cost-based value, such as low cost. The second retention form is called value-added retention which suggests retention is derived from the value of customer service. If the client is unhappy, this will have an adverse effect on the quality of customer service. In Bolton et al. [22], the research verified that value-based retention is the outcome of a cognitive comparison method where cognitive assessment occurs before the affective reaction occurs.

The first form of customer retention has been referred to for this research, which focuses on customer retention as a result of service quality where offering value to clients is extremely satisfying to clients on an ongoing basis [23]. Generally, in previous research in multiple service industries, the immediate connection of service value was recognised as a reliable predictor of customer retention and compatible with previous and present research [17, 18, 24, 25].

Recognised from previous results about important relationships for these two constructs, the current research is restricted to continuing research in this regard. The results of this research in the context of clothing store brands may lead to current information about this relationship. The first hypothesis was proposed as follows on the basis of the discussion above: *H1: Service value has a positive effect on customer retention.*

2.3 Service quality

Service quality is frequently described as a discrepancy between service expectations and perceived service provided by the organisation and employee service performance [26]. In the early establishment of the notion of service quality, the [20] service quality model, also known as the Nordic view, recognised two dimensions of service quality, namely, technical quality linked to "what customers get" and functional quality linked to "how they get it." The research by Behera defines technical quality as "what the customer receives as a consequence of relationships with a service company" and functional quality, defining it as "the transfer of technical quality." Behera also found that the company's corporate image was constructed by the technical and functional quality of service.

In his research on the mid-end model, Parasuraman et al [27] described service quality as a worldwide judgement or attitude pertaining to the general excellence or superiority of the service and Parasuraman et al [27] again quoted this concept. Edward [20] claim that Yee et al. [28] define service quality as being the most commonly accepted for studying service quality by other academics. This research uses the definition of Parasuraman et al [27].

2.3.1 Relationship between service quality and customer retention

The link between service quality and customer retention has become a focal point in literature about services [6, 29]. Service quality plays a vital role in achieving a competitive advantage by providing high-quality service capable of achieving customer retention and shaping the enterprise's beneficial result, such as customer loyalty and reducing company rivals [30].

Some of the recent research has provided coherent evidence on the immediate and beneficial relationship between service quality and retention of customers, such as studies carried out by Ranaweera, Venetis and Han [6, 29, 31]. These findings were in line with the following past studies Ennew and Hennig [32, 33].

Several scientists studied the immediate impact of customer retention on the connection between each dimension of service quality. For instance, Islam et al. [34] provided quantitative results on service quality delivery and its impact of customer retention in Malaysia's banking sector, which disclosed that the dimension of certainty, empathy, reliability, and responsiveness has a connection to but it does not have an important impact on customer retention. Only tangible dimensions have a favourable connection and an important effect on the retention of customers. The results of the assessment are not in line with [35] results obtained by the Malaysia Islamic banks using PAKSERV, taken from the SERVQUAL scale where tangible, assurance, honesty, customisation and formality dimensions have an important connection with customer retention but not reliability. Another Hume and Sullivan [30] research of the public health service discovered that the dimension of certainty, compassion and responsiveness has important customer retention relationships, they are, however, not tangible and reliable. While the research by Islam et al. [34] discovered that all SERVQUAL sizes were important for customer retention, the outcome showed that the tools used to evaluate the quality of service were extremely accurate and valid. Inconsistent results from the impact of SERVQUAL sizes on customer retention in previous research may, however, be due to cultural differences [34].

Studies applying a higher order construct of service quality are growing due to the complexity of abstract service quality. For example, studies of Rajaratnam et al. [36] in rural tourism destinations in Malaysia used seven dimensions of service quality, i.e. availability and logistics, key tourism experience, hygiene, data, safety, value-for-money and hospitality through formative strategy using the structural equation model methodology. Their research results indicate that the quality of service was important for the retention of tourists, and suggests that the quality of service was a direct precedent for customer retention. They also verified that the quality of service is a multidimensional structure. While the research of mobile communications providers in China by Daniel et al. [37] adjusted the hierarchical model of service quality as suggested by Brady [38].

Although there have been several approaches to evaluating service quality in the aforementioned literature, such as multi-dimensional service quality, one-dimensional service quality, and hierarchical service quality model, service quality remains an important building block for customer retention. This is due to the nature of the quality of service and the connection between client retention being seen as linear, which shows that high quality of service results may result in elevated customer retention. The current research proposes the following hypothesis in order to meet the current gap in clothing store brands in China: *H2: Service quality has a positive effect on customer retention.*

2.3.2 Relationship between service quality and service value

There is no doubt that marketing scientists have long been interested in service quality and service value.

The notion of service quality was described as the evaluation of the general excellence or superiority of the service by the customer [27]. While Cronin et al. [16] conceptualised the quality of service that reflected the performance-based assessment of the perception of service quality during a service meeting. They described perceived service quality in an article by Zeithaml et al. [39] that consisted of elements through performance, expectation and disconfirmation. Functional quality, such as response, reliability, empathy, certainty and technical quality (in tangible terms) becomes a major driver for customers in evaluating the value of purchasing a product or service in terms of cost, effort, emotion, connection and social aspects. The client will perceive the high-quality output, speed of service delivery, comfort and friendly services through the superiority of service provided by the supplier as a significant effect on the client compared to what they are giving.

Service quality therefore plays a significant role in determining the value of service. Results from the research conducted by Cronin et al. [16] demonstrate that perception of quality mainly has a defined service value where it highlights quality rather than price associated with its exchange transaction. Other scholars, including De Oña and Mazzulla [40], reference past studies, such as Lai and Petrick [41], Muala and Ngo and Nguyen [42, 43] and later research, in which comparable findings consistently showed that the customer's perceived service value assessment was directly dependent on the customer's perceived service quality assessment. Ledden et al. [17] acknowledged that several scholars like Ganguli and Roy [44], Wang, Shieh, and Hsiao, [45] and Erdil and Yildiz [46] regarded quality as a value component dimension. Sweeney and Soutar [47] also stressed that the function of service value was a crucial component of decision-making, and perceived service quality was a major antecedent of service value.

In distinct contexts, such as clothing store brands, further research of this connection enriches current understanding. The third hypothesis for this connection was thus proposed as follows: *H3: Service quality has a positive effect on service value.*

2.3.3 Service value as mediator between service quality and customer retention

The function of service value is essential in the relationship between the service provider and the client. In addition, the function of service significance is substantial and unique where this variable can serve as a mediator [16] and moderator [48]. Having a better knowledge of the role of service value is therefore essential.

In the connection between service quality and customer retention, service value was empirically recognised as a mediating variable [15, 49]. A study conducted by Cronin and Brady [16, 38] suggests that the customer retention evaluation was preceded by a cognitively focused service quality and service value evaluation. According toChen and Yang [49], service value serves as a more important predictor in service evaluation than service quality. Empirical research findings by Kuo et al. [50] state that the overall impacts of post-purchase intention contributed to the value of service, followed by the quality of service and customer retention. It has been found that providing a greater service value will boost customer's favourable behavioural intent and word of mouth.

Research using a structural equation model conducted by Hume and Mort [30] in performing arts settings in Australia illustrates the importance of the quality of service to customer retention, while intention to repurchase was fully dependent on service value, but not the peripheral quality of service and evaluation of emotions. They suggest that executives should concentrate on core quality of service as a main factor, such as showing or acting in performing arts, to determine intention to purchase again. Their research also discovered that the quality of service, peripheral quality of service and evaluation of emotions was directly connected to the value of service, but were not important for customer retention and service value. The complicated mediation function of service value should therefore warrant further studies.

Research by Kwun [51] is another proof of the mediation role of service quality. His research of a campus food service discovered that the role of service value mediation varied from gender to gender. The connection between service quality and customer retention was fully mediated by service value for female customers, while the quality of food and menu was partly mediated by the importance of service. On the contrary, only food quality was mediated by male consumers' service value. The outcome shows that evaluations produced by male and female customers on the characteristics of campus food service gave mixed trade-off advantages on the quality of service and had distinct impacts on satisfaction and customer behaviour.

The results of the research by Kwun [51], Kuo et al. [50] and Cronin et al. [16] indicate that quality of service, value of service and retention of customers can jointly contribute to a significant impact on customer service intention and perception after purchase. They also suggest analysing the integration of these factors through a multivariate analytical strategy.

The aforementioned literature review endorsed the function of service value as a mediating variable between the service quality relationship and the retention of customers. Continuous inquiry into this relationship will lead to current understanding due to the sort of service and place variables that may be uncertain in the relationship finding. Since the analysis of this connection has been ignored specifically in the context of the Chinese clothing store brands, the issue remains. To fill the current gap, it would therefore be useful to present the research to find a response on this relationship. Based on the empirical evidence, this research suggested the fourth hypothesis as follows: *H4: Service value mediates has a positive effect on the service quality and customer retention.*

2.4 Research framework

Building on past empirical research, the article presents a model for finding out the relationship between service quality and customer retention in clothing store brands: Mediating effect of service value (Figure 1). Furthermore, the model indicates a number of hypotheses that support these suggested backgrounds.

2.5 Research methodology

This chapter addressed the following problems pertaining to research design, such as population, sample size, sampling technique, study hypothesis, questionnaire design, method of assessment, and reliability outcome. The research population measured who is buying at clothing store brands in China. About 385 questionnaires had valid answers and were used in this research paper for data analysis; the information was analysed using partial least square regression. According toSekaran (2003), a total of 385 answers were used and subsequently analysed, resulting in a response rate of 80% using the 5-point Likert scale for all answers with (1 = strongly disagree, 2 = disagree, 3 = undecided, 4 = agree, 5 = strongly agree). The questionnaire is split into four components: section (1) population variables (8) items; section (2) quality of service measurements (22) items from Zeithaml et al. [39]; section (3) service value (5) items from Ledden et al. [17]. Finally, section (4) adjusted (4) items from Han [31] for customer retention. In conclusion, the researcher used convenience sampling processes in social science studies as a prevalent type of sampling design (thorough sampling). Mohr and Spekman [52] provide an appropriate database for scientists to use the methods of statistical inference. This sampling design strategy is also relevant in the marketing of services.

3 Results and discussion

3.1 Profile of respondents

The aim of the profile of respondents is to study their characteristics according to the study samples that were established. In terms of gender, men accounted for 38.7% clothing consumers, while women accounted for the remainder of 61.3%. It was determined that men are less inclined to buy clothing store brands than women. Analysis by age provided information about the purchasing behaviour of people. It was shown that older people have a higher propensity to purchase clothing store brands.

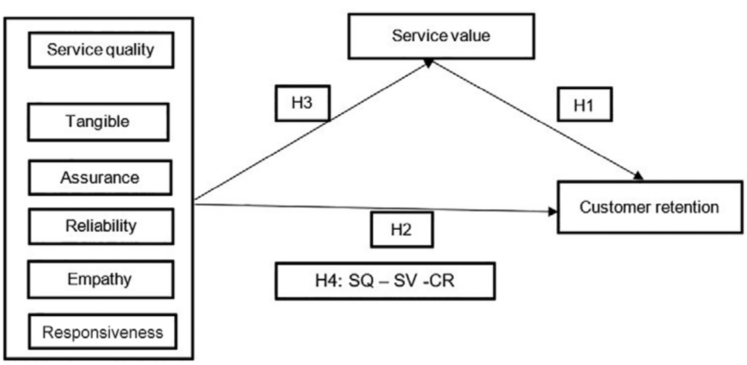

Figure 1: Research framework

This study explains demographic characteristics of the respondents. It shows that respondents aged 36 to 45 years account for 30.6% of the total. The customer of a clothing store brand is represented by consumers who earn a monthly salary of between €1,001 and €1,500, or 24.7% of the total. The selection of clothing brands is highly dependent on the information available to the buyer. Clients with higher qualifications will be choosier in their consumption decisions. The data for the qualification level show that the highest level achieved by the largest group of respondents is master degree, which accounts for 55.1%. At 51.2% of the respondents are married, 32.7% are single, while 16.1 are other. The occupation of consumers also has an effect on their consumption behaviours. Features of respondents by occupation subjugated by government equal 12.9%. The number of respondents bought clothing brands non- government equal 15.7%, while the majority of respondents bought clothing brands 39.9% own employee, 18.8% have bought clothing brands by student and 12.7% have bought clothing brands by others.

Table 1: Demographic characteristics of participants

Demographic	Characteristic	Frequency	Percentage
Gender	Male	236	61.3
	Female	149	38.7
Age	Below 25	72	18.7
	25–35	89	23.1
	36–45	118	30.6
	46–55	42	10.9
	56 and older	64	16.6
Monthly salary (EUR)	Below 500	79	20.5
	500–1000	85	22.1
	1001–1500	95	24.7
	1501–2000	83	21.6
	Above 2001	43	11.1
Qualification	Diploma	15	3.9
	Degree	54	14
	Master	212	55.1
	Doctoral	104	27
Marital status	Single	126	32.7
	Married	197	51.2
	Other	62	16.1
Occupation	Government	51	13.2
	Non- government	59	15.4
	Own employee	154	39.9
	Student	72	18.8
	Others	49	12.7
Source of information	Online	272	70.6
	Friends	42	10.9
	Relatives	14	3.6
	Advertisement	24	6.2
	Others	33	8.6

Table 2: Correlations among variables and discriminant validity

	ASS	CR	EMP	REL	RES	SV	TAN
ASS	0.901						
CR	0.799	0.832					
EMP	0.891	0.832	0.899				
REL	0.863	0.799	0.887	0.888			
RES	0.885	0.786	0.895	0.885	0.902		
SV	0.842	0.843	0.867	0.821	0.835	0.886	
TAN	0.876	0.780	0.876	0.758	0.871	0.812	0.906

Note: ASS = assurance; CR = customer retention; EMP = empathy; REL = reliability; RES = responsiveness; SV = service value; TAN = tangible

The results show how the respondents obtained their source of information about clothing store brands. Several response options were made available and the respondents were allowed to choose more than one option. A large portion of the respondents (272 respondents) obtained source information about clothing store brands using online services and a total of 42 respondents received source information about clothing store brands from friends. Meanwhile, 14 respondents obtained their source information about clothing store brands via their relative(s). Lastly, 24 of the respondents got their source information by advertisement and 33 of the respondents received their source information about clothing store brands from others.

3.2 Discriminant validity

The degree to which items distinguish between constructs or measure separate ideas is the discriminating validity of the measures. Hair et al. [54] explained in this regard that the discriminating validity stipulates that the average variance extracted from each latent construct (AVE) should be higher than the highest square correlation of the other latent construct as recommended by Fornell and Cha [55] criterion, and that the loading of the item should be greater than all its cross loading.

3.3 Testing of hypotheses

First of all, for direct hypothesis, H1 postulates an important connection between service value and customer retention where previous studies frequently support this relationship. The connection between these relationships also discovered an important aspect in the context of clothing store brands service ($\beta = 0.467$, S.E. = 0.090, t = 5.174, $p < 0.000$) in the same manner. In addition, R^2 was found to be 0.754 in customer retention and was substantially explained by the value of the service. All service value products played key roles in shaping service value construction and thus revealed the significant role of quality service value results in building customer retention relationship.

Confirmed and approved the second assumption predicting an important connection between SQ and CR. The regression outcome produced by SmartPLS showed that the link between SQ and CR was important ($\beta = 0.430$, S.E. = 0.094, t = 4.593, $p < 0.000$). The β value was comparatively large with the $t > 2.58$. Another statistical finding is that the R2 for CR was 0.704, which was near to the significant variance rate accounted for by SQ. All variables under SQ were discovered to portray SQ build considerably on the basis of Table 3. Compared to other factors in SQ, the outer weights of 0.105 and t = 4.664 for EMP had reached the largest value. This shows that EMP was the most important element in the development of service quality in the context of clothing store brands.

In previous studies, the connection between SQ and SV was not always linked with a fresh idea in marketing research. SQ was discovered to be considerably combined with SV ($\beta = 0.876$, S.E. = 0.025, t = 35.084, $p < 0.000$) and R2 value of 0.768 as shown in Figure 1. Therefore, this study's third hypothesis was verified and adopted. SQ variables coded as TAN, REL, RES, ASS and EMP had important features in molding SQ build that caused important connection between SQ and SV which were found to be more fruitful.

Finally, the hypothesis of indirect impact predicts that SV will mediate the connection between SQ and CR. In previous research, there has been numerous evidence of the role of SV as mediator between these two factors. SQ has a substantial direct impact on CR with a route coefficient of $\beta = 0.430$

in the current research. The indirect impact of SV was 0.409 and statistically substantial with t = 5.120 ($p < 0.000$) after the mediating variable was inserted for regression. The path coefficient β was decreased to 0.021 for direct impact between SQ and CR but still has an important impact as shown in Table1. Determining the amount of mediation and the outcome shows that SV has a complete mediation impact on the connection between SQ and CR in the next assessment. Therefore, it was verified and approved in the H4 hypothesis.

According to Hair [40], the primary assessment criterion of the structural model by PLS-SEM is the R^2 measures and to determine the significance level of the path coefficients. The reason is because the objective of the prediction-oriented PLSSEM approach is to explain the variance of endogenous latent variable and reasonably high R^2 value should be obtained. A rule of thumb in marketing research studies, R^2 values of 0.75, 0.50, or 0.25 for endogenous latent variables in the structural model can be represented as substantial, moderate, or weak, respectively. Accordingly, the obtained R^2 value can be used to interpret the quality of the structural model which indicates the explanatory variance by the exogenous variables contained in the endogenous variable. Assessment results it can be explained the R^2 was found to be 0.754 for CR, indicating that SQ can account for 75.4% of the variance in the CR, which was substantial level.

3.4 Discussion

The finding of all hypotheses validated and verified the complete mediation of service quality and client retention. With the presence of service value as a mediator between service quality and client retention, the value of R^2 improved from 70.4% to 75.4% for variance strength explained in client retention. Recognised service value has a complete mediation impact between service quality and customer retention, the size of the indirect impact for service value consequence. In the past, studies on the role of service

Table 3: Summary of hypotheses testing results for direct and indirect effect

Hypotheses	Path	β	S.E.	t-value	p-value
H1	SV → CR	0.467	0.090	5.174	0.000
H2	SQ → CR	0.430	0.094	4.593	0.000
H2a	ASS → CR	0.081	0.018	4.548	0.000
H2b	EMP → CR	0.105	0.023	4.664	0.000
H2c	REL → CR	0.099	0.022	4.568	0.000
H2d	RES → CR	0.082	0.018	4.518	0.000
H2e	TAN → CR	0.082	0.018	4.578	0.000
H3	SQ → SV	0.876	0.025	35.084	0.000
H3a	ASS → SV	0.166	0.007	23.915	0.000
H3b	EMP → SV	0.215	0.006	35.250	0.000
H3c	REL → SV	0.202	0.007	29.801	0.000
H3d	RES → SV	0.167	0.005	33.285	0.000
H3e	TAN → SV	0.167	0.005	32.270	0.000
H4	SQ → SV → CR	0.409	0.080	5.120	0.000
H4a	ASS → SV → CR	0.078	0.015	5.053	0.000
H4b	EMP → SV → CR	0.100	0.020	5.020	0.000
H4c	REL → SV → CR	0.094	0.018	5.123	0.000
H4d	RES → SV → CR	0.078	0.015	5.208	0.000
H4e	TAN → SV → CR	0.078	0.015	5.125	0.000

Note: CR = customer retention; SV = service value; SQ = Service quality; EMP = empathy; REL = reliability; RES = responsiveness; ASS = assurance ; TAN = tangible value as a mediator are not a new topic. Numerous studies provided the same results on the impact of service quality mediation such as Kwun [51] in Food Services, Chen et al. [49] in Taiwan Financial Services, [56] in clothing Services, Hume and Mort [30] in Australian Art Performance, Cronin et al. [16] in Service Environments and Brady et al. [38] in Fast Food Services in America and Ecuador.

A complete mediation impact between service quality and customer retention in the environment of clothing store brands in China is the most compelling reason for service value because service quality has a powerful direct impact on service value and customer retention. Customers are constantly seeking quality in the services they have engaged in and are always a key component in the delivery of marketing and business services. High service quality establishment will result in high service value [57] and customer retention [31, 58, 59] and attracting another opportunity to increase the picture of an organisation [12, 60], encourages beneficial behaviours such as reuse intention, positive word of mouth referral and allegiance [18]. By comparison, poor quality service leads to the damage of a company's strength owing to adverse reaction, bad word of mouth and low client buyback [9]. In brief, there will be trash in the trash [54].

This research demonstrates that all dimensions of service quality worked intensively together to serve value and retention in the clothing store brands environment. Equally relevant to this research, it was observed that the measurement of quality of service in clothing store brands is better by combining functional and technical factors rather than functional aspects alone as proposed by [20]. As a quality output reaches an acceptable level, after engaging in the service, clothing store brands clients will evaluate the benefit versus disadvantages. Because clients prefer high quality in clothing store brands, perception of value becomes beneficial and minor defects that clients encounter during service consumption are ignored. This is obvious in the current research where quality performance variables in service value continued to achieve the highest effect rating, followed by value and social variables, although several elements of service quality were discovered to be poor. This finding coincided with the research by Cronin et al. [16]. Therefore, it was suggested that clothing store brands operators should concentrate on and promote any initiative to enhance quality in clothing store brands services from the moment to the time to deliver high value to their clients, leading to retention of customers. Possible initiatives such as cashless clothing payments, mini retail outlets or valet services can be regarded for value-added services to increase an excellent experience among clothing store brands customers and should guarantee precious characteristics known to the client. High customer worthiness, after service usage, will make a specific service unique that could enhance company competitiveness through elevated level of retention. This declaration was backed by the current research results where customer-represented service comparison had attained the highest loading in the retention structure compared to other parts. In addition, the results of this research consistent with the research by [16] where they discovered that retention was mainly explained by quality of service and value of service and further conclude that cognitive evaluations precede emotional reactions. Their research also shows that joint attempts are being made to enhance quality, value and retention as a means of refining perceptions of client service. In addition to suggestions, the current research also highlights manager's requirements to decide the correct approaches, embed clear direction among employee and frequent tracking to guarantee excellence in delivering value to their clients [49].

4 Conclusion

The aim of the research is to explore the service quality and service value effect on customer retention to clothing store brands in China. The study's descriptive outcome indicates that there is still a mild level of customer retention on service in clothing store brands. This implies the perception on medium level of service quality and service price of clothing store brands clients. The research framework structural assessment shows that the model has appropriate predictive significance in PLS-SEM technique for the constructs through the blindfolding procedure. Briefly, the findings of the study show the difference in the effect of service quality. The connections between service quality, service value and retention with fairly elevated statistical results were discovered to be important for an immediate impact. This demonstrates that the quality of service and the value of service are efficient variables in the retention of customers.

Service value had shown complete mediation impacts in subsequent assessment studies. In short, it has discovered acceptance of four hypotheses developed from the study framework. Thus, the study's research goals were achieved.

The research results were discussed and suggested that executives integrate workable approaches in clothing store brands in terms of mixing quality and value of service in order to provide favourable retention reactions among clothing store brands clients. Periodically evaluating customer feedback on services can help service providers improve their clothing store brands service and monitor any changes in behavioural trends that serve as inputs to further improve clothing store brands services. There have been acknowledged several constraints in the study that offer possibilities in future studies. Future studies were proposed to include intentional conduct, real behaviour, other antecedent factors and research model moderator variables. Application of the low and high order construct notion in future research is also proposed. Further validation of the results of the study was promoted through the expansion of sample size, building type, geographic region and other service industries.

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Characteristics, Protection and Functional Design of Three-Layer Laminate for Medical Footwear
Značilnosti, zaščita in funkcionalna zasnova trislojnega laminata za medicinsko obutev

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Abstract
The aim of this paper is to determine the influence of the washing and sterilization process in real hospital conditions on the microbial barrier properties of textile laminate used in medicine for protective clothing. The paper focuses on the functional design of three-layer laminate for medical footwear in surgery and in rooms where aseptic working conditions are required. The permeability and durability of the microbial barrier were determined after 0, 10 and 20 washing and sterilization procedures according to previously developed methods. Bacterial endospores of apathogenic species of the genera *Geobacillus stearothermophilus* and *Bacillus atrophaeus* were used. A functional design of the protective shoe cover was proposed. The results showed an extremely effective microbial barrier and the durability of the sample after 0, 10 and 20 washing and sterilization procedures, and over a period of one, two and three months.
Keywords: microbial barrier, three-layer laminate, medical footwear, functional design

Izvleček
Namen članka je bil ugotoviti vpliv pranja in sterilizacije v realnih bolnišničnih razmerah na mikrobiološke barierne lastnosti tekstilnega laminata, ki se uporablja za medicinska zaščitna oblačila. Predstavljena je funkcionalna zasnova trislojnega laminata za medicinsko obutev v operacijskih dvoranah in drugih prostorih z aseptičnimi delovnimi razmerami. Prepustnost in obstojnost mikrobne bariere sta bili določeni po nič, desetih in dvajsetih pranjih in postopkih sterilizacije po predhodno razvitih metodah. Uporabljene so bile nepatogene bakterijske endospore iz rodu Bacillus, Geobacillus stearothermophilus in Bacillus atrophaeus. Predlagana je bila funkcionalna zasnova zaščitnega pokrivala za čevlje. Rezultati po nič, desetih in dvajsetih postopkih pranja in sterilizacije ter v obdobju enega, dveh in treh mesecev odležanja vzorcev so pokazali izjemno učinkovito in obstojno mikrobno bariero.
Ključne besede: mikrobna bariera, trislojni laminat, medicinska obutev, funkcionalna zasnova

1 Introduction

Textile laminate is a two- or multi-layer textile material that is connected with a polymer layer to form a whole. The substrate on which the polymer layer is applied can be woven, knitted or non-woven fabric. Polyurethane (PU) is a multi-purpose coating polymer used to coat protective clothing [1, 2]. The laminate properties are a combination of the properties of the base fabric and the polymer layer. The result of such a combination provides many properties that the individual components cannot provide. The substrate or base fabric gives the composite material mechanical strength and carries the coating layer applied to it. High-quality coated fabrics require high-quality base woven and knitted fabrics [1–5]. The polyurethane (PU) polymer layer gives the laminate the property of liquid impermeability and the possibility of water vapour transfer from the body into the environment [6–8]. Due to the comfort of the microporous structure, which allows breathing, and the possibility of passing through the sterilization medium, polyurethane (PU) is an acceptable material for a wide range of medical applications [9, 10]. Medical textiles belong to the group of technical textiles and include all textile products used in medicine, gowns, caps, medical footwear, etc. Conditions of application, i.e. multiple washing and sterilization damage the textile material, which limits its use [11]. In order to meet medical standards, reusable medical textiles must, with regard to the purpose, meet some of the basic conditions, such as being impermeable to microorganisms, being biocompatible, having the possibility of chemical and thermal sterilization, ensuring the stability of shape and dimensions; having the possibility of rational and economical production, etc. [12]. Washing or dry cleaning and sterilization allow their reuse. Reusable medical textiles are more cost-effective on account of cost reduction. How long they are used depends solely on the efficiency of the microbial barrier and mechanical properties where they are needed [13]. The function of medical textiles is often to protect against bacteria and viruses originating from staff and patients. They must be free of toxic ingredients, maintain integrity and durability, and withstand the physical conditions of standard stress during use [2, 12]. Manufacturers do not recommend woven fabrics and knits as an adequate barrier for use in medicine if they are single-layered and without surface treatment because their pores are far larger than microorganisms. However, woven and knitted fabrics are still the most commonly used textile materials for medical purposes [14, 15]. Polyurethane (PU) is one of the specific polymeric materials widely used in various fields and even in medicine for medical synthetic materials and dressings due to its exceptional properties, such as softness, comfort to touch and long-lasting pressure, and balance of mechanical properties, especially when sandwiched between textile fabrics [16]. By selecting the appropriate properties of the components in the layers of the laminate, exceptional properties can be achieved that can satisfy a variety of medical applications.

The aim of this research was to determine the influence of washing and sterilization on the permeability and durability of the microbial barrier of a three-layer laminate PES (woven fabric)/PU/PES (knitted fabric) for strong and durable medical footwear. Changes were identified after 0, 10 and 20 washing and sterilization processes under real hospital conditions. The durability or retention time of the microbial barrier of sterilized, diagonally packed laminate packages (EN ISO 11607-1 2009) was determined after storage over a period of one, two and three months under real hospital conditions [17]. The aim of this work was also to create a functional design of a three-layer laminate for medical footwear.

2 Experimental part

2.1 Materials and methods

A three-layer textile laminate made of PES/PU/PES and produced at the company Čateks in Čakovec, Croatia (Table 1) was used. The front side of the fabric sample is woven in plain weave, while the back side consists of knitted fabric. There is a polyurethane layer between the woven and the knitted fabric. The samples shall be used for medical purposes.

Table 1: Basic parameters of the three-layer PES/PU/PES laminate

Sample	Composition	Structure	Mass per unit area (g/m^2)	Thickness (mm)
PES/PU/PES	1st layer	100% PES woven fabric, plain	213.84	0.42
	2nd layer	100% PU		
	3th layer	100% PES knitted fabric, tricot		

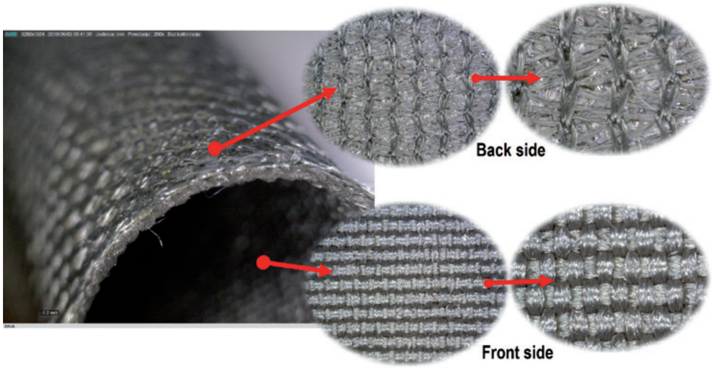

Figure 1: Surface view of the PES/PU/PES laminate

Figure 1 shows the surface of the three-layer PES/PU/PES laminate from the front and back side.
The washing and sterilization procedures were carried out in the specialized laundry unit of the Clinical Hospital Centre Zagreb – Rebro. The sample was washed in a continuous washing machine (JENSEN brand) according to a specially defined procedure (see Table 2) [18]. The sample was sterilized in a Selectomat PL MMM hospital steam sterilizer (Münchener Medizin Mechanik) at 134 °C and at a pressure of 2.5 bar for five minutes. The samples were sterilized after each washing cycle.

Table 2: Washing parameters

Washing solution	0.7 g/kg Ce; 2.5 g/kg Ca
Disinfecting agent	4 g/kg Cc
Temperature (°C)	60
Bath ratio	1:5

Commercial names of all products are not given due to the confidentiality of the participating laundry and the impartiality of the research. Ca – polycarboxylate (< 5%), sodium hydroxide (10–20%). Cc – ethoxylated fat alcohol < C15 & < 5 EO (25–30%), solvent, 2-propanol, methanol (0.1–0.25%), amphoteric surfactants (1–2%), additives (0.1–0.25%). Ce – formic acid (50–100%).

2.2 Microbial barrier properties

Samples are packed in sterilization packages and sterilized at 134 °C for five minutes (number of the samples: 18). Microorganisms are then applied to the samples under aseptic conditions by rubbing on the sample surface. The application of microorganisms is followed by incubation for 24 hours. After incubation, prints are taken with a CT3P agar plate. First from the back side, then from the front side. A 72-hour incubation at 35 °C is then performed, followed by the counting of bacterial colonies (CFU) [19].
Figure 2 shows a schematic representation of the testing of microbial barrier properties.
The bacterial spores of the genus *Geobacillus stearothermophilus* and *Bacillus atrophaeus* were used as the only dry-type microorganisms. The use of a suspension moistens the fabric and the permeability is altered [18].

2.3 Microbial barrier durability testing

The tested samples (22 cm x 22 cm) were packed in packages after 10 and 20 washing cycles (Package number: 60). Gauze was packed into each package, under which absorbent paper "Whatmann No. 1" of 1 cm2 was placed. The packed packages were sterilized (134 °C for five minutes) and placed in a protected

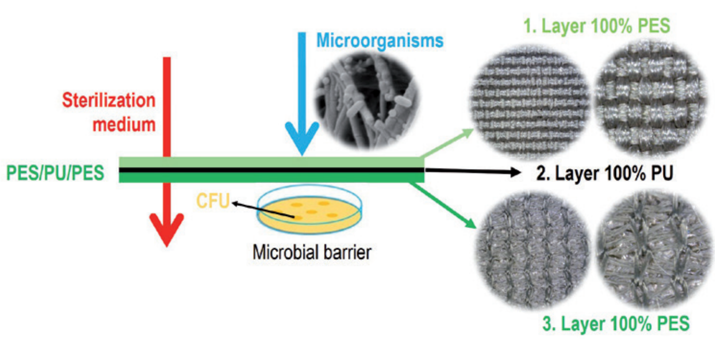

Figure 2: Microbial barrier properties testing

warehouse with a temperature of 15–30 °C and a relative humidity of 30–60%. The storage time of the packages was one, two and three months. After the specified time, the packages were removed from the warehouse and unwrapped under sterile conditions. Using sing tweezers, the absorbent paper was removed from the package and placed in a test tube with a Brain-Heart broth. After incubation for 48 hours at a temperature of 35 °C, a change in the visually clarity of the broth was observed, i.e. whether turbidity has occurred. The sterility was additionally checked by fitting on a solid nutrient basis, while 5 ml of Brain-Heart broth was planted on an agar containing 5% sheep blood. After 48 hours of incubation at 35 °C, the number of bacterial colonies was observed [19].

3 Results and discussion

Due to the washing and sterilization procedures, a three-layer textile PES/PU/PES laminate shrinks. This, in turn, results in an increase in fabric thickness. A change in mass per unit area and thickness was determined according to the relevant standard and is shown in Table 3 [20, 21].

Table 4 shows the number of microorganisms on the front and the number of microorganisms passed through to the back. The number of microorganisms on the surface represents the number of microorganisms remaining on the surface of the sample after bacterial endospores of pathogenic species of the genus *Geobacillus stearothermophilus* 10^5 and *Bacillus atrophaeus* 10^6 were rubbed with a stick.

In the PES/PU/PES sample, there was a 3.8-fold increase in the number of microorganisms absorbed on the surface (283 CFU) compared to the initial values (74 CFU). However, despite the growth of microorganisms on the front of the sample, there was no leaking of microorganisms on to the back. It can be concluded that the increase in retained microorganisms on the surface layer is due to the rough surface after 20 washing and sterilization cycles. One of the reasons for the impermeability of the microbial barrier is the polyurethane layer present in the sample. The rougher surface has the ability to retain a larger number of microorganisms, which is evident from the results obtained (see Table 4). The durability of the microbial barrier was determined using the method of sterilized diagonally packed packages (one layer; EN ISO 11607-1: 2009) after storage for one, two and three months. The results of the testing of the durability and efficiency of the microbial barrier over one, two and three months after a series of 10 and 20 washing and sterilization procedures are presented in Table 5.

The testing results of the durability of the microbial barrier over a period of one, two and three months show a satisfactory durability of the microbial barrier of the textile laminate. There was no contamination of the contents of the package. The impermeable microbial barrier is very important for use in medicine and other sterile areas.

Table 3: Results of the design parameters of the three-layer laminate

Sample	Washing and sterilization cycles	Mass per unit area			Thickness		
		Mean (g/m^2)	SD[a)] (g/m^2)	CV[b)] (%)	Mean (mm)	SD[a)] (mm)	CV[b)] (%)
PES/PU/PES	0	213.84	2.43	1.14	0.42	0.01	1.76
	10	217.44	1.56	0.72	0.45	0.01	3.17
	20	214.12	2.47	1.16	0.44	0.01	1.29

[a)] *standard deviation;* [b)] *coefficient variation*

Table 4: Microbial barrier test results

Sample	Number of isolates	Washing and sterilization cycles	The average number of bacterial colonies on the front side (CFU)	The average number of bacterial colonies on the back side (CFU)
PES/PU/PES	6	0	74	0
	6	10	240	0
	6	20	283	0

Table 5: Results of microbial barrier durability testing after one, two and three months, and after 10 and 20 washing and sterilization procedures

Sample	Washing and sterilization cycles	Testing after (months)		
		1	2	3
PES/PU/PES	10	NMR[a)]	NMR	NMR
	20	NMR	NMR	NMR

[a)] *no growth in microorganisms in the package*

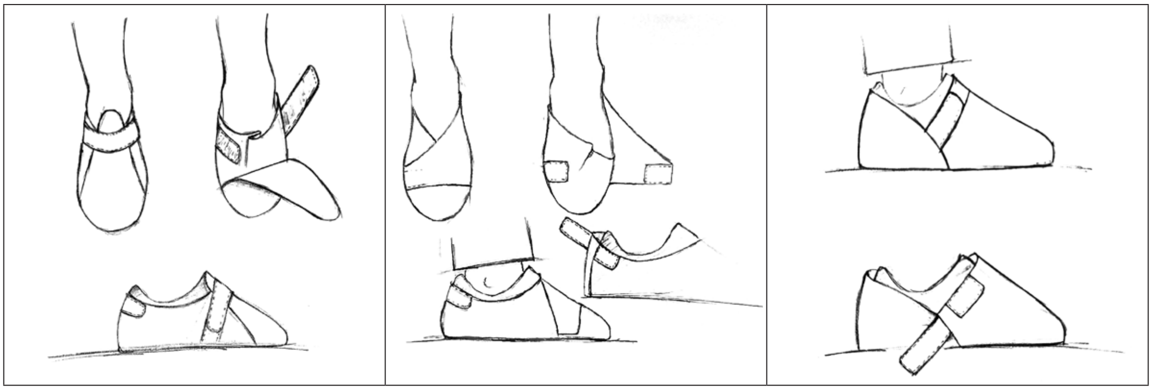

Figure 3: Functional design of three-layer laminate for medical footwear

Figure 3 shows the functional design of a three-layer laminate for medical footwear. The design was created so that it is easy to put it on and take off, and the aesthetic component as added value was not neglected. The models shown in Figure 3 are more demanding to produce than ordinary disposable covers. They have a tread to which the upper part of the cover is sewn, which is larger than the foot or shoe. To adhere better to the foot, they are fitted with Velcro strap so that they can be worn over shoes or on bare foot, as can be seen in the examples. Three design solutions of a three-layer laminate cover to be used for microbial protection are proposed in Figure 3.

Proposal a) can be easily pulled on and adjusted with the Velcro strap on the heel, while the excess material is folded forward, then covered with an accessory and secured with the Velcro strap. In example b), the heel is adjusted in the same way as in the previous model and the Velcro strap is fastened at the front to stand firmly around the foot. Similar to slippers, the model in example c) is only fixed on the side in the middle where the front and back are joined. In this way, it is more flexible and, as shown, can be easily adjusted and tightened with the Velcro strap. The proposed design is somewhat more expensive and demanding to produce, but since it is not a disposable product, it would be worthwhile to invest a little more at first. The models presented can be adapted according to needs and requirements, and it is proposed to produce them in two sizes to improve fit and therefore protection. This is also a good example of sustainability and environmental protection, as reusable covers also reduce waste, which is another added value.

4 Conclusion

This paper investigates the efficiency of the microbial barrier of textile PES/PU/PES laminate with respect to washing stability and the sterilization process. Bacterial spores of the genus *Geobacillus stearothermophilus and Bacillus atrophaeus* were used. The durability of the microbial barrier over a period of one, two and three months after 10 and 20 washing and sterilization procedures was determined for a three-layer textile laminate. The results showed that the sample had an effective microbial barrier through 20 washing and sterilization procedures. The durability of the microbial barrier tested over three months also showed that there was no penetration of microorganisms to the inside of the sterilized packaging. From this it can be concluded that the sample has an extremely effective microbial barrier and its durability is guaranteed after 10 and 20 washing and sterilization procedures, and after storage under strictly controlled conditions for a period of one, two and three months. The PU membrane between two polyester layers in the laminate has a major influence on this impermeable barrier. The value of retained microorganisms on the surface of the three-layer PES/PU/PES laminate was 3.8 times higher than the initial value. The process of washing and sterilizing samples often leads to their permanent shrinkage, and these changes cause a linear increase in mass and thickness.

A functional design of a three-layer laminate for medical footwear with emphasis on flexibility, practicality and wearing comfort was proposed. All three proposals meet both the functional and aesthetic value.

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Comfort Evaluation of Cyclists Jerseys Using Wear Trial Test
Vrednotenje udobnosti kolesarskih dresov s testom poskusnega nošenja

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Abstract
The aim of this study was to evaluate the wear comfort of four commercially available cycling outfits and understand various subjective parameters of garments through consumer perception, which will enable the design and development of an optimized outfit. A questionnaire was developed specifically to address various key aspects such as tactile sensation, garment fit with reference to size, garment assembly, garment aesthetics (style and shape), comfort (before, during and after wearing) and overall satisfaction (relating to design of the garment and style). Three outfits were fabricated from polyester fabric and one from polyamide/elastane (80%/20%) fabric. They were assessed by four male professional cyclists (age 22–25) at various stages of a test protocol of 45 minutes total duration, of which 20 minutes was flat cycling. The four tested garments showed greater differences between the sensorial comfort perceptions than thermophysiological comfort. The sensorial comfort sensation was found to be mainly correlated with fabric properties, fit, construction techniques and moisture sensation, whereas the thermophysiological comfort was found to be affected by the fabric characteristics, the test environment conditions and level of activity. Additionally, manual measurements showed great brand-based differences between garments of the same specified size M (medium). Overall, the polyamide/elastane jersey was perceived as a better cycling outfit than the polyester outfit. The results of this study provide guidance for the optimal design and development of professional cyclist outfits.
Keywords: cycling garment, sensorial comfort, thermophysiological comfort, subjective wear trial

Izvleček
Cilj raziskave je oceniti udobnost nošenja štirih tržno dostopnih kolesarskih oblačil in razumevanje različnih subjektivnih parametrov zaznavanja oblačil potrošnikov pri uporabi, kar bo omogočilo oblikovanje in razvoj optimiziranega oblačila. Izdelan je bil poseben vprašalnik za obravnavo različnih ključnih vidikov, kot so občutek otipa, prileganje oblačila glede na velikost, sestavljanje oblačila, estetika oblačila (slog, oblika), udobje pred, med in po nošenju ter splošno zadovoljstvo, povezano z dizajnom oblačila in slogom. Tri obleke so bile izdelane iz poliestrske tkanine, ena pa iz mešanice poliamida in elastana (80 %/20 %). Ocenili so jih štirje moški poklicni kolesarji (stari od 22 do 25 let) v različnih fazah testnega proto-

kola, ki je skupaj trajal 45 minut, od tega je bilo 20 minut kolesarjenja po ravnem. Štiri preizkušena oblačila so pokazala večje razlike v otipu kot v toplotnofiziološkem udobju. Ugotovljeno je bilo, da je čutno udobje v glavnem odvisno od lastnosti tkanine, prileganja oblačila, konstrukcijskih rešitev in občutenja vlage, medtem ko na toplotnofiziološko udobje poleg značilnosti tkanine vplivajo razmere v preskusnem okolju in stopnja aktivnosti. Poleg tega so meritve pokazale velike razlike v dimenzijah med oblačili različnih blagovnih znamk, a enake velikosti M (srednje). Na splošno je bil za kolesarje bolje ocenjen dres iz mešanice poliamid/elastana kot dres iz poliestra. Rezultati te študije dajejo smernice za optimalno zasnovo in razvoj dresa za poklicne kolesarje.
Ključne besede: kolesarsko oblačilo, senzorično udobje, toplotnofiziološko udobje, subjektivno poskusno nošenje

1 Introduction

Clothing comfort is an essential aspect of users' performance and is taken into consideration as a quality characteristic while choosing a particular garment [1]. Clothing comfort is, however, an extremely complex subject and is the result of many interactions between physical, psychological, and physiological factors [2–4]. Sports apparel not only requires comfort, but also functionality. At the same time, these garments must have excellent thermophysiological properties adapted to a particular sport discipline [5]. Thermophysiological comfort, also referred to as thermal comfort, is crucially important for sportswear worn next to skin, where rapid heat transfer, moisture vapor and liquid moisture transfer from skin to the outer fabric surface is required [2]. These factors are influenced by the thermophysiological conditions of the human body [6–8].

Cycling is one of the most popular sports and can be performed in many different weather conditions. Therefore, the expectations that cyclists have in terms of the comfort of athletic apparel have increased. Clothing comfort includes all the comfort sensations produced by a garment [1, 9, 10]. Many studies have been conducted in relation to cycling clothing, in particular taking into consideration ergonomic issues and the effect of compression on performance and recovery [11–13]. Other fields of research cover injury reduction [14, 15], the design of cycling clothing [16, 17], and aerodynamic behaviour and various other aspects of comfort [18–23]. However, previous studies showed that cycling apparel requires further investigation.

Comfort can be a psychological state, a physical sensation or both simultaneously [24]. Most importantly, the development of clothing should consider the anatomical features of individuals (anthropometric data), and biomechanical and functional features (skills and physical limitations while performing occupational or sport activities) [25]and hence tend to be complex and iterative. These factors can overlap and correlate significantly with the subjective evaluation performed and provided by users, especially regarding usability, wearability and safety.

Clothing designed specifically for certain functionalities (i.e. a cycling garment worn next to the skin) has been shown to cause heat stress, and reduce the task efficiency as well as the range-of-motion of the wearer [26]. The process of design therefore begins by first establishing the many requirements of the user. An extra concern for cyclists is low back pain, the most prevalent injury and a problem for their health [26–29], and several garments have been developed to assist with fatigue and improve motor function. However, athlete compliance is likely to be affected due to the discomfort and inconvenience of these garments.

A wear trial deploying various evaluation techniques was set up to investigate the functional and comfort requirements of users. The findings of comfort need and the effects of various garment attributes from different wear trials will provide insight into the design and development of proper garment criteria that are required to satisfy an athlete's critical ergonomic needs, and acting upon these insights will eventually improve their performance. The purpose of this study was to quantify the wearers' perceived comfort responses to existing cycling garments in order to identify the influential garment attributes.

2 Materials and methods

2.1 Materials

2.1.1 Test garments

In this study, four commercially available cyclist outfit garments were obtained from A.S. Adventure Ghent, Belgium. All samples were short-sleeved, medium size T-shirts/jerseys. The selected garments were differentiated by fabric composition and structure as shown in Table 1.

Table 1: Fabric composition and structural parameters of selected garments A–D

Garment code	Fibre composition	Garment size	Fabric structure	Courses (cm)	Wales (cm)	Thickness (mm)	Air permeability (mm/s)
A	100% PES[a)]	M[d)]	1x1 rib	20	19	0.40	929.5
B	100% PES	M	Interlock with 1x1 rib	25	18	0.44	1,515.0
C	80% PA[b)]/20%EL[c)]	M	1x1 rib	24	16	0.53	1,150.0
D	100% PES	M	1x1 rib with 3D knitted	20	16	0.69	1,262.5

[a)] polyester; [b)] polyamide; [c)] elastane; [d)] medium

2.1.2 Test subjects

Four male professional cyclists aged between 22 and 25 years from Bahir Dar, Ethiopia were selected to participate as human subjects in the wear trial test of the study. All subjects were healthy volunteers who exercised regularly. Each subject was given one experimental garment over a given time span.

The participants were informed beforehand about the scope of the test, procedure and risks [31, 32]. Informed consent was signed by all subjects, but they were not informed about the details of the clothing materials in order to avoid any influence on their subjective ratings. However, subjects were invited to have a pre-trial before formal trials to determine their individual cycling intensity and understanding of the questions and the procedures involved.

2.2 Methods

2.2.1 Fabric characterization

Fabric analysis was performed on the four different styles of purchased jerseys, including fibre compositions, knit structure, stitch density, thickness and air permeability. The thickness of the fabrics was measured according to ASTM D1777 using a MESDANLAB Digital thickness tester. The air permeability properties of the fabrics were measured using an FX 3300 air permeability tester according to the ISO 9237 standard with a 100 Pa air pressure difference and a 20 mm^2 test area.

2.2.2 Garment design and size measurement comparison

Garment design: To determine the recommended fit, the sizing charts provided by each retailer were taken from the relevant websites [33–37]. These charts stated the recommended size of the wearer at the chest for a small, medium and large size sample. These were observed further to assess the significance of the measurements recorded and garment assembling for the selected samples.

Garment size measurements: Each sample was measured to highlight differences in garment size and shape, according to the four brands A, B, C and D.

2.3 Wear trials

2.3.1 Subjective assessment of comfort

A variety of methods is typically applied to assess comfort in trials. Some studies use a combination of methods, including one or more questionnaire items. Likert-type rating scales and numeric rating scales have been used [38, 39]. Of these scales, some were oriented to assess “comfort” and “discomfort”, while some were bipolar [13, 14, 19, 37]. In this study, Likert rating scales with different scales were used to assess the subjective perception of the subject. Likert scaling is a unidimensional scaling method useful when measuring latent constructs, i.e. the characteristics of people, such as attitudes, feelings and opinions.

2.3.2 Environmental conditions and test protocol

To gather data about parameters affecting the thermal comfort status of the test persons, temperature, wind speed and relative humidity measurements were recorded objectively (Table 2). The measurements were carried out using the mobile app Live weather forecast widget, which provides daily weather forecasting. All tests in the scope of wear trials were conducted in actual working field environments from 6 am to 9 am, when the sun is still very low, in order to limit the effect of solar radiation. The experimental protocol was approved by Bahir Dar University, Ethiopian Institute of Textile and Fashion Technology Institutional Review Board (IRB) (10th November 2018).

Test subjects followed an exercise protocol consisting of four activities for 45 minutes: the subjects first wore the T-shirt and then they rested with it for 5 minutes in the test environment prior to the conducting of the next test. The subjects then warmed up by doing stretching for 10 minutes according to their normal

Table 2: Environmental conditions during the field trial

Test day	Outside temperature (°C)	Relative air humidity (%)	Wind speed (km/h)	Avg. cycling speed (km/h)
1st	17	74	1.1	27.8
2nd	16	96	0	25.4
3rd	10	48	1.8	29
4th	10	48	0	29.5

stretching routine. Next, the subjects started cycling trials consisting of a 20 minutes flat ride, followed by cooling down (recovery) for 10 minutes (see Figure 1).

2.3.3 Response and validation

We used the rating system described by Wong et al. [41, 42] and a specially designed questionnaire, as well as an assessment scale defined by ISO 10551:2004 [43] and ISO 7730:2005 [44]. At the end of each trial phase, each participant was asked about their psychological state and thermophysiological comfort, and this was recorded by rating thermal comfort and sensations, such as moisture perception, thermal sensation, and overall physiological and psychological comfort during the cycling period.

The first evaluation was made during the initial touch of the fabric, during the first minute when the subjects handled and wore the garment. During exercise, subjective ratings of comfort and discomfort of the T-shirts, broadly relating to thermal and tactile experience, were recorded. The subjects were instructed at each questioning to concentrate on the area of their upper bodies. The explanation of and judgment between the various sensations and the rating scale were discussed with subjects in advance of the experiments. After each trial, the subjects were asked to compare the overall comfort of the four tested T-shirts they had worn for the trial and restate their preference. The rating scales are shown in Table 3.

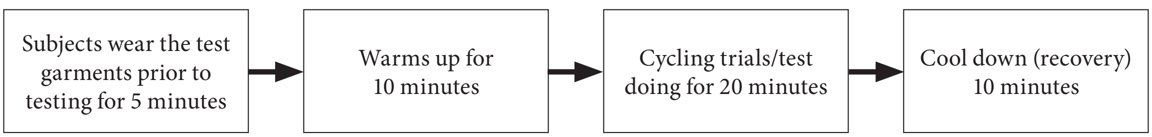


Figure 1: Flow diagram of exercise protocol

Table 3: Rating scales

Comfort	Evaluation criteria	Scale	Remark
Psychological	Clothing size fit	5-point scale	1 (too loose) … 5 (tight fit)
	Stretchiness	9-point scale	1 (very stretchable) … 9 (non-stretchy)
	Overall garment look	9-point scale	1 (like very much) … 9 (dislike)
Thermal	Skin sweat sensation	5-point scale	1 (neutral) … 5 (extremely wet)
	Skin temperature sensation	7-point scale	1 (cold) … 7 (hot)
Sensorial	Stiffness and sticky sensation against the skin	9-point scale	1 (not at all) … 9 (extremely strong)
Ergonomic	Easy of body movements while cycling with ensemble	5-point scale	1 (very stiff) … 5 (very flexible)
	Level of ease in performing duties	7-point scale	1 (very easy) … 7 (very difficult)
	Degree of comfort	9-point scale	1 (extremely uncomfortable) … 9 (extremely comfortable)
	Overall fit of ensemble for the purpose	7-point scale	1 (very poorly) … 7 (very well)

2.3.4 Statistical analysis

IBM SPSS 21 statistics and Microsoft Excel software were used to analyse the results. Coefficient of variation and mean were used to quantify the variation of various subjective, physiological and objective comfort parameters.

3 Results and discussion

3.1 Fabric characterization

All fibre compositions were taken directly from the care label. Samples, A, B and D were made of polyester and sample C was made of a combination of polyamide and elastane. Polyamide is a strong fibre that has excellent elastic recovery behaviour after stretching [45]. These properties are very important and crucial for (compression) sportswear garments due to the frequent strain on the fabric during use (wearing and washing). Polyester, on the other hand, is characterized by maintaining the stability of its structure, and offering excellent heat resistance and good moisture transport properties. It does not easily extend and has a low cost [46]. However, in the case of garments that require stretching, nylon is better than polyester, while polyester is favoured over nylon for maintaining stability. Fabric thickness, air permeability, structure and stich density of fabrics A–D are presented in Table 1. Fabric (A) has the lowest air permeability value (929.5 mm/s) but is the thinnest fabric. Air permeability varied significantly between fabric A and fabric B (1,515 mm/s), with thicknesses of 0.44 mm and 0.40 mm and different structures, respectively. Fabrics A and C contained different compositions of 100% PES (fabric A) and 80% PA/20% EL (fabric C), with a 1x1 rib structure. The 1x1 rib with 3D knitted sample D was the thickest (0.69 mm) and demonstrated lower air permeability than fabric B and a lower fabric density than samples A, B and C.

3.2 Garment design and size measurement comparison

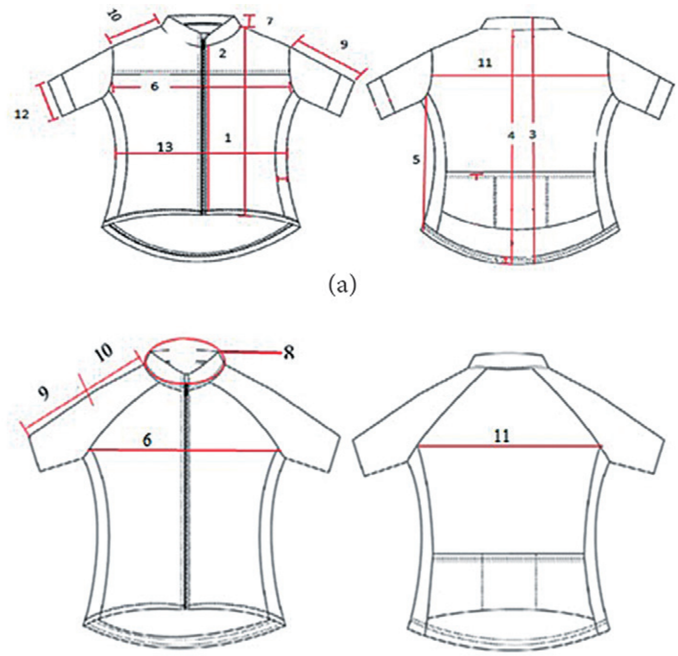
Design detail and size measurements were compared for the four brands of test garment purchased. There were variations in the design in each type. Detailed features of each garment sample are shown in the Figure 2.

a) b) c) d)

Figure 2: Photo of sample garments A (a), B (b), C (c) & D (d)

3.2.1 Garment size measurements

Each sample was measured to highlight differences in garment size and shape, according to the four brands A, B, C and D. Figure 3 shows the points at which the samples were measured and Table 4 details the manual measurements (cm) taken for the four samples. The measurements listed show variations between ready to wear samples of the same size (medium).



(a)

(b)

Figure 3: Measurement points of sample garments: a) A, D; b) B, C

Table 4: Measurements variation of garment samples A, B, C, D

Serial number	Measurement point	A (cm)	B (cm)	C (cm)	D (cm)	Mean ± SD[a)] (cm)	CV[b)] (%)
1	Full length front	63.5	63.5	59.5	63	62.4 ± 1.9	3.10
2	Centre front length	54	54	52	53	53.3 ± 1	1.80
3	Back full length	76.5	71	70	77.5	73.8 ± 3.8	5.15
4	Centre back length	72.5	68	66	73.5	70.0 ± 3.6	5.12
5	Side seam length	45	46	43	46	45.0 ± 1.4	3.14
6	Across chest (seam to seam) front	46	49	45	48	47.0 ± 1.8	3.88
7	Collar stand length	41	42	40	47	42.5 ± 3.1	7.32
8	Collar stand width (neck circumference)	4	3	4	4	3.8 ± 0.5	13.33
9	Sleeve length	24	23.5	35	35	29.4 ± 6.5	22.12
10	Shoulder length	12.5	13.5	14	9	12.3 ± 2.3	18.41
11	Across back	42	42	40.5	47	42.9 ± 2.8	6.62
12	Cuff length straight (1/2)	14.5	13.5	11	14	13.3 ± 1.6	11.73
13	Waist length front	46	44	40	44	43.5 ± 2.5	5.79

[a)] standard deviation; [b)] coefficient of variation

This variations in the measures of the different brands for what should nominally be the same medium size are remarkable. The size seems to be derived from the same recommended chest size and waist size (centre front length has a CV of 1.8%, full front a CV of 3.1% and across chest a CV of 3.88%). The large CV for other measures (for example CV of 11.73% for cuff length) is thought to affect the fit of the garment. In particular, the chest size measure affects the pressure distributed by the garments when worn, especially if the wearer of the garment is towards the upper limit of the suggested size measurement.

These variations in measurement between the sample garments illustrate the need for more detailed sizing recommendations for users to ensure correct fit and consequently sufficient compression. It is also believed that these variations in grading could affect the pressure distributed across sizes. It must be taken into consideration that only one medium size sample was measured per brand. This helps to highlight the differences between garments when consumers purchase them.

Generally, it should be noted that while significant differences in grading were highlighted by these measurements, only one sample of one size was examined. Therefore, some of the measurements taken may be unrepresentative as a whole and the result of mistakes in production. The relationship between the size of the garments and the fibre content will again be of interest when looking at the pressure distribution of the samples. Where the samples have the same recommended torso size but show varying chest measurements, the effect of this on the compression will also be highly interesting. Therefore, further research to investigate these differences in grading on a much larger number of samples may be helpful.

3.3 Subjective assessment of comfort perception during cycling

3.3.1 Psychological responses

Subjects were required to assess the overall look, stretchiness and clothing fit by handling and putting the garments on respectively.This was their initial preference of the sample before starting the exercise. Out of the four samples tested (Figure 4), assessments of the perceived stretchability/non-stretchability property of the garments generally fell in the "neutral" category (4–5) for sample C, B and D. In fact, these three samples are not similar by fabric type (such as fabric structure, fibre composition and thickness), as shown in Table 1. Therefore, the assessment of stretchy/non-stretchy did not differ significantly by structure, fibre composition or thickness, and provides evidence that the perception of garment stretchability is not affected solely by fabric type.

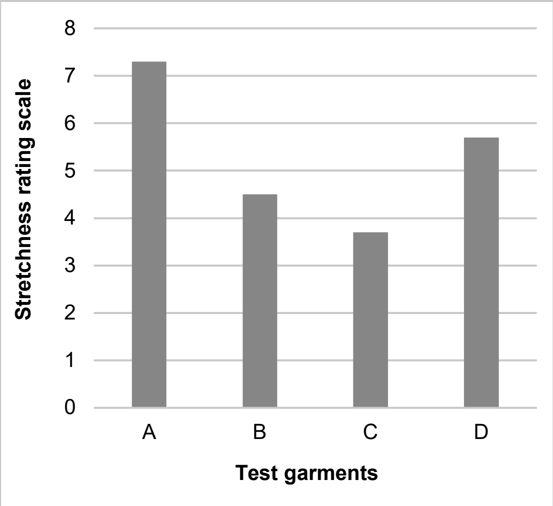


Figure 4: Average value of stretchiness, 9-point scale: 1 (very stretchable) … 9 (non-stretchy)

In addition, for sample A, the fabric stretchability was rated "moderately non-stretchy", while it was rated as "loose" in terms of tightness/looseness of the garment fit to the body. This agrees with garment size measurements (Table 4), which are above the average for almost all measurement points considered. Similarly, the same "loose" fit assessment was given to garment D, while garments B and C were rated as "normal/moderate" (Figure 5). The fabrics of these garments were different in terms of composition and

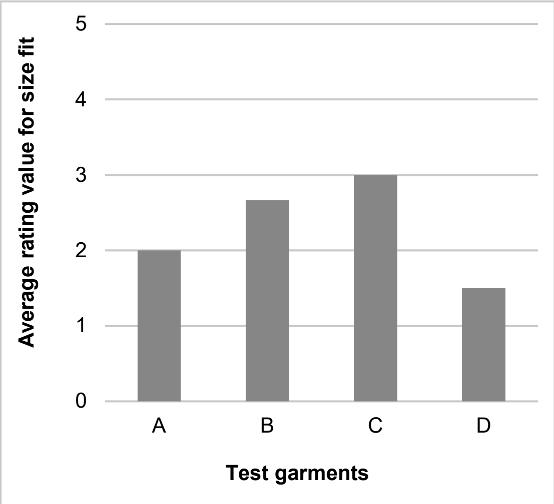


Figure 5: Average value of clothing size fit, 5-point scale: 1 (too loose) … 5 (tight fit)

other properties (Table 1), and it is therefore unlikely that this minor difference in fit could have contributed to greater discomfort during wear. The results further indicated that garment C constructed from polyamide and elastane material (Table 1) is liked more than the other samples (Figure 6).
We concluded from the pre-test ratings of the psychological responses "clothing size fit", "stretchiness" and "overall look" that the polyamide-elastane garment C was more accepted than the polyester garments A and D, and slightly more accepted than garment B, which coincides with the slightly better fit of garment C than garment B, while garments A and D were on the loose side. Garment B was, however, considered more stretchy than garment C, so a good fit and adequate stretching contribute to better acceptance. Thus, the difference between the garments observed on these subjective dimensions under pre-test conditions may be due to the characteristics of the fabrics from which the garments were constructed, as well as the design, assembly and overall appearance/look of the ensemble.

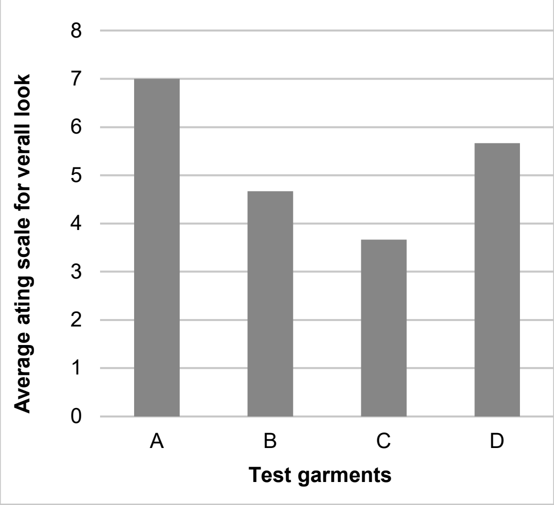


Figure 6: Overall look: how well the garment is liked/disliked, 9-point scale: 1 (well liked) … 9 (disliked)

Thermal transmission is thought to be one of the most important factors affecting clothing comfort [47, 48]. The thermal insulation of clothing is affected by many physical factors, such as fabric thickness, the amount of body surface area covered by the garment, garment design (looseness and tightness) and number of fabric layers [46].
The subjective measurements were collected during field trials during the warmup and cycling immediately after recovery stages. The test data was split and grouped over the first two days 1 and 2 and last two cold/dry days (Table 2) in order to show whether the environment influences the results or not. All clothing trials were performed in the actual working field environment (cold and warm) at an average temperature of between 10 °C and 16.5 °C, a relative humidity of between 48% and 85%, and a wind speed of 0.9 and 0.6 km/h, respectively. The average age, height and weight of the subjects were 22.8 ± 1.0 years, 173.8 ± 10.7 cm and 61.6 ± 4.5 kg, respectively as described in Table 1. Each subject tested all four of the garments on separate occasions.

Thermal-sweat sensation: Professional cyclists train much more intensively, and the wetness level and expectations are therefore completely different for recreational cyclists. Physiological effects during different activity levels (such as seated, exercising and recovery condition) of the test were mostly related to moisture properties (Figures 7–9). The different garment fabrics did have effects on thermal perception and comfort, as well as on the moisture related perceptions of the wearer.
The various subjective thermal-wet sensations changed in different ways during exercise under different climatic condition. Figures 7 and 8 illustrate the results. Most of the garment-related moisture sensation increased significantly with activity (Figure 7), but the warm skin temperature sensation (Figure 8) showed a decreasing trend over time in the start/recovery stage. In general, we see from the mean skin temperature of the test subjects while wearing the test

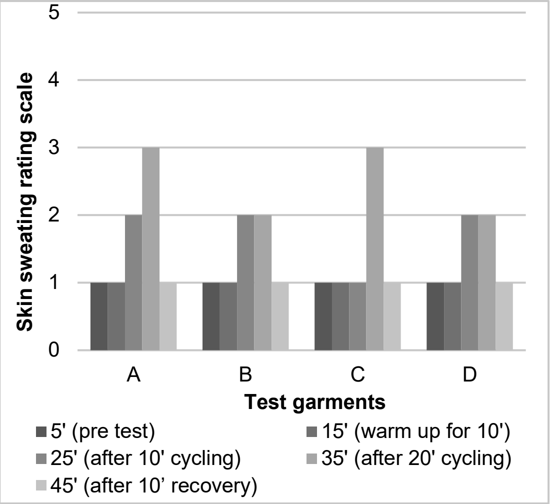


Figure 7: Average skin sweat sensation of test subjects while wearing analysed test garments under different activity level, 5-point scale: 1 (neutral) ... 5 (extremely wet)

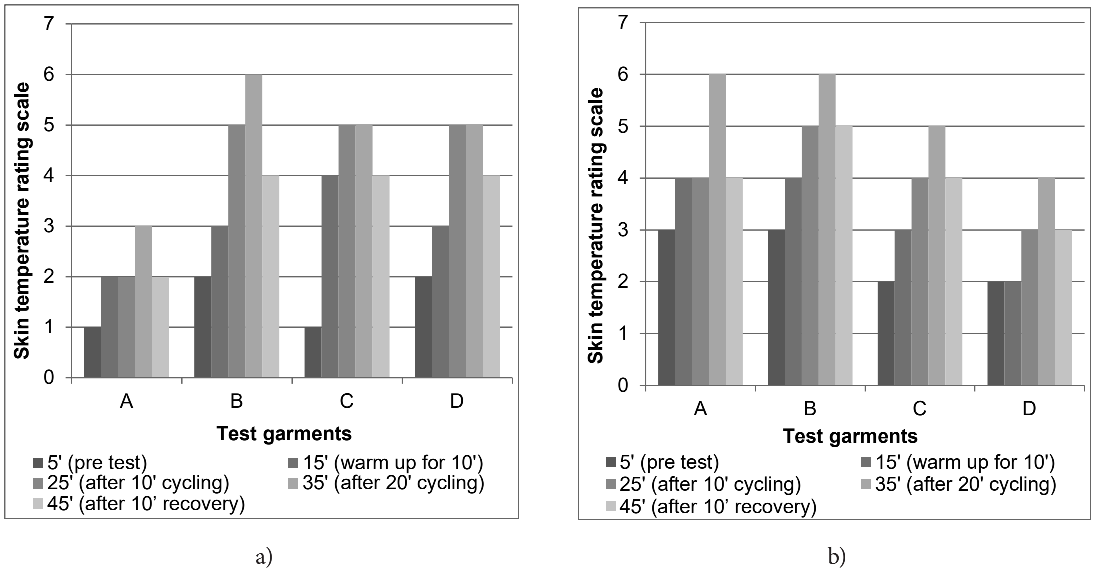


Figure 8: Mean skin temperature sensation of test subjects while wearing analysed sample test garments under different activity level: a) in cold and b) in warm climatic conditions; 7–point scale: 1 (cold) ... 7 (hot)

garments in the warm and cold climatic conditions (Figure 8 a–b) that garment B resulted in greater perception of heat in both conditions, and garment A resulted in greater perception of skin wetness than garment B, but less in heat sensation in cold conditions after 20 minutes of cycling. The subjects had similar neutral skin temperature sensation while wearing garments C and D in cold conditions (Figure 8 a) and they indicated less skin wetness after 20 minutes of cycling while wearing garment D than while wearing garment A (Figure 7).

From these results, we can also deduce that the loose garments A and D result in lower skin temperature sensation in hot conditions during recovery, with the loosest garment D resulting in the lowest skin temperature perception overall in warm conditions. However, for aerodynamic reasons, cyclists want to avoid loose garments. Among the good fitting garments B and C, garment C demonstrated the best temperature properties (i.e. lowest skin temperature sensation in warm conditions), but a higher sweat sensation rating after 20 minutes of cycling than garment B. This showed that the polyester garment B had a lower moisture uptake from the skin than the polyamide/elastane fabric C. It is important to note that even during the warmup, garment A was perceived as cold in cold conditions, while this was the case for garment D in warm conditions, demonstrating that a looser fit results in more training activity required to warm up. Considering the deviation from the neutral 4 scale in skin temperature sensation during the warmup and cycling phases, garment C performs best in cold conditions (Figure 8a), followed by garment D.

Sensorial comfort: With regard to skin contact attributes in terms of the perceived sticky sensation of the skin, garment A and B were assessed as moderately sticky, one score higher than C, and two scores above the loosest garment D (i.e. where less fabric comes into contact with the skin) as shown in Figure 10. Not much variation was identified between the garments in terms of stiffness, with all recording a score close to the value of 4, meaning all give a moderately stiff touch sensation.

Ergonomic comfort: Considering the degree of comfort, garment B rated as neutral (score of 5) whereas garment C was rated as very comfortable (score of 8) and was also perceived as normal (score of 3) for ease of body movements while cycling (Figure 9) and making it easier (score of 2) to perform duties. To a lesser extent, the less stiff garment B (Figure 10) was also perceived as somewhat easy for performing duties (score of 3). When we compared overall fit for the purpose of the garment, garment D was assessed as fitting poorly for the desired purpose (score of 2) and difficult to perform the task (score of 4).

These differences in the skin feel sensations of the garments, combined with the perceived pre-test differences among the garments for “feel” and “comfort”

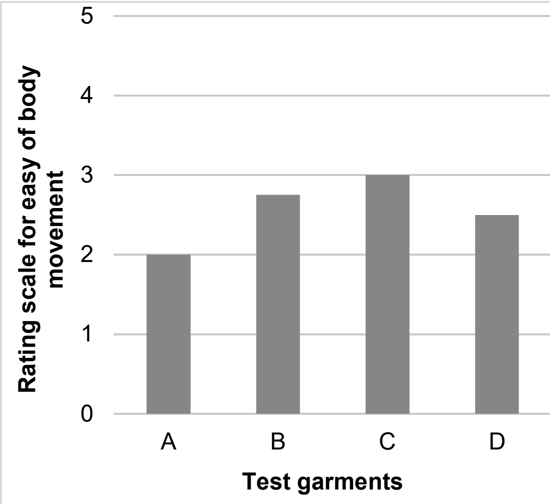


Figure 9: Average value of ease of body movements while cycling with ensemble, 5-point scale: 1 (very stiff) … 5 (very flexible)

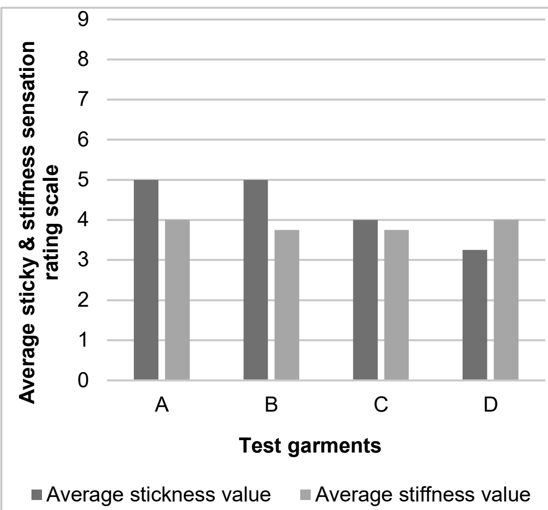


Figure 10: Average stickiness and stiffness sensation against the skin for the duration of whole activity phase, 9-point scale: 1 (not at all) … 9 (extremely strong)

suggest that the tactile characteristics of the fabrics contributed, along with the moisture and thermal sensations, to the overall assessment of the comfort of the garments during the study. The overall findings for the comparative comfort of the garments were consistent with the expected response that the considered polyamide/elastane fabric (C) tends to be more comfortable, with excellent elasticity and recovery behaviour, while polyester fabrics A-B-D are more likely to produce discomfort. This assumption was made during a pre-test questionnaire in which the subjects generally expressed the most favourable opinion regarding the 80/20 polyamide/elastane fabric (C) and the least favourable opinion regarding the 100% polyester fabric (D) with respect to fabric-skin contact sensation. This pre-test also ensured that all garments, regardless of the fabric from which they were constructed, fit the participants equally well in various body areas. Adapting the polyester garment construction in such a way that it has a good stretchability and can thus be made to fit tighter (garment B) is highly preferred by the cyclists over the other polyester fabrics (A and D), but nevertheless remains less preferred than the polyamide garment, with a higher stickiness, lower fit and higher skin temperature sensation. Though the sweat perception of fabric B was better (lower) than fabric C after 20 minutes of cycling, this brings less weight for cyclists who expect a certain level of sweat during sport [30].

4 Conclusion

Significant brand-based differences between garments of the same specified size M were observed and overall, the polyamide/elastane jersey was perceived as the best. The results suggest that thermal and moisture sensations of different T-shirts primarily relate to the different physiological state of subjects (i.e. perception of skin temperature and wetness). On the other hand, tactile sensations were found to differ between the subjects wearing different jersey, whilst differences in these sensations did not change over time (exercise), nor show any significant difference between warm and cold conditions. It therefore seems that the tactile and fit sensations were mainly determined by fabric-skin-contact, not by the environmental conditions or exercise. This suggests that the overall preferences of the subjects for clothing worn next to the skin, in both thermal conditions of these trials, were mainly determined by the tactile and fit sensations and not by the thermal-wet sensations. The result shows that sensations of comfort-discomfort in clothing worn next to the skin can be influenced by several factors, including the environment and the physiological state of the wearers, as well as the type of fibre used in manufacturing the fabrics and garment fit. The interaction between the factors is also important, and overall acceptability of a garment is not easily predicted by simple handling tests. The cyclists do seem to prefer tight fitting garments with enough stretch. The results of this study provide guidance for the optimal design and development of cyclist outfits.

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Effect of Hairiness on Fabric Colour Characteristics
Vpliv kosmatosti tkanine na njene barvne značilnosti

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Abstract
Designing the colour appearance of textiles requires taking into account their surface properties, hairiness among others. The villi protruding on the surface not only affect the quality of textile dyeing, but also largely determine its optical properties and the colour phenomenon. The analysis of studies of optical properties shows that the influence of hairiness on the phenomenon of colour is not well understood and that the amount of hairiness at which colour changes become significant for human perception remains indefinable. In this work, we studied the change in colour characteristics depending on the change in the hairiness of woollen fabrics, comparing "yarn – raw fabric", "yarn – raised fabric", "raw fabric – raised fabric". Hairiness was estimated by the hairiness index, which was obtained from the analysis of sample microphotographs of yarn and fabric using software. The value of colour characteristics (lightness, chroma and colour difference) was measured in the CIELAB colour space (1976) using a spectrophotometer. The obtained experimental results showed that the changes in lightness and saturation of textile materials from the index of its hairiness are directly proportional. However, the value of changes is different for raw and napped fabrics, undyed and dyed samples, the initial colour hue and raw material composition also making certain adjustments. This study analysed the colour difference and established the level of variation in hairiness at which the colour mismatch between woollen fabrics becomes visually noticeable. The results of the study can be used to predict the colour and design the optical properties of fabrics for weaving and finishing.
Keywords: woollen fabrics, colour, hairiness index, colour characteristics

Izvleček
Oblikovanje barvnega videza tekstilij zahteva upoštevanje njihovih površinskih lastnosti, med katerimi je tudi kosmatost. Resice, ki štrlijo s površine, ne vplivajo samo na kakovost barvanja tekstilije, temveč v veliki meri določajo tudi njene optične lastnosti in značilnosti barve. Analiza študij optičnih lastnosti kaže, da mehanizem vpliva kosmatosti na značilnosti barve ni dobro razumljen, obseg kosmatosti, pri katerem postanejo spremembe barve pomembne za človekovo zaznavanje, pa ni nedoločljiv. V tej raziskavi so bile proučevane spremembe barvnih lastnosti zaradi spremembe kosmatosti volnenih tkanin, in sicer s primerjavo „preja – surova tkanina", „preja – kosmatena tkanina" in „surova tkanina – kosmatena tkanina". Kosmatost je bila ocenjena z indeksom kosmatosti, ki smo ga dobili z računalniško analizo mikrofotografij vzorcev preje in tkanin. Vrednost barvnih lastnosti (svetlost, kroma in barvna razlika) smo izmerili v barvnem prostoru CIELAB (1976) s pomočjo spektrofotometra. Dobljeni eksperimentalni rezultati so pokazali, da so spremembe svetlosti in nasičenosti tekstilnega materiala neposredno sorazmerne z indeksom kosmatosti tekstilije. Vrednost sprememb je različna za surove in kosmatene tkanine, nebarvane in barvane vzorce, prav tako pa dodatno vplivata začetni barvni ton in surovinska sestava. V raziskavi je bila analizirana barvna razlika in določena stopnja

variacije kosmatosti, pri kateri postane barvna neskladnost med volnenimi tkaninami vizualno opazna. Rezultate te študije lahko uporabimo za napovedovanje barve in oblikovanje optičnih lastnosti tkanin pri tkanju in plemenitenju.
Ključne besede: volnene tkanine, barva, indeks kosmatosti, značilnosti barve

1 Introduction

The regular change of popular colours and their shades is relatively typical of the modern fashion industry; therefore, compliance with the trend colour is one of the key properties of products that ensure the commercial success of the textile production. Nevertheless, reproducing the desired colour in a textile product is a complex challenge, which includes not only choosing the optimal ratio of the dye formulation, but also predicting the appearance of colour in the texture of the finished material [1]. Texture features need to be taken into account since the nature of reflection or absorption of light rays depends on the material roughness, which affects perception and consequently distorts the colour phenomenon. Based on the requirements for the colour design and appearance of the fabric, attention needs to be paid to the structure when dyeing, hairiness being of particular importance. On the one hand, the layer of villi affects the adhesion of the dye, increasing the hydrophobicity of the textile [2–6]. Since this leads to an increase in the contact angle, to ensure the quality of dyeing, hairiness is undesirable [7]. On the other hand, the hairiness layer significantly affects the appearance of the finished product, as it is the main surface property [8]. Studies [9–13] show that reflection, gloss, lustre, dichroic, birefringence, as well as the lightness of the fabric surface largely depend on the density of villi and their orientation, i.e. hairiness affects the phenomenon of colour [13]. The influence of hairiness on dyeing textiles is much more studied than on the phenomenon of colour. Although studies [14–17] show that hairiness increases the lightness of the surface, these results are not sufficient to determine the colour of fabrics, since the amount of hairiness at which the changes in lightness become significant for human perception remains uncertain. Therefore, predicting the colour of textiles, especially for fuzzy fabrics such as fleece or flannel, continues to be difficult.

It is worth noting another difficulty in designing colours for textiles that are made from dyed yarn and are nap. The difficulty lies in choosing the appropriate colour of yarn, which should correspond to the established standard for the colour of fabric after all stages of its production and processing. The aim of this research was to study the effect of hairiness on colour indices in fabrics made from undyed and dyed yarn with a different amount of hairiness.

2 Experimental

2.1 Materials

Samples of yarn and fabric from the assortment of the Vladi textile enterprise (Kharkov, Ukraine) [18], made from woollen fibres, or from a mixture of woollen and chemical fibres (cf. Table 1), were selected as the subject of the study, since hairiness is especially

Table 1: Structural characteristics of textile samples

Textile set	Fabric composition	Weave pattern	Fabric weight (g/m^2)	Fabric density (threads/dm)
S1	100% acrylic	plain	280	160
S2	90% wool, 10% acrylic	plain	250	120
S3	100% wool	3/2 twill	270	160
S4	80% merino wool, 20% polyester	2/4 matting	250	170
S5	60% wool, 40% polyester	2/2 twill	270	110
S6	40% wool, 5% merino wool, 35% acrylic, 20% polyester	2/2 twill	275	120
S7	50% wool, 30% polyester, 20% acrylic	2/3 twill	245	180
S8	70% wool, 30% polyester	2/2 twill	250	160

a woollen characteristic and can easily be increased by the raising process. All samples were divided into 8 sets, each of which included yarn, a raw fabric made from this yarn and the same fabric after the raising process (cf. Table 1). Each set was prepared in two versions, i.e. undyed and dyed. Undyed samples had a natural white shade of wool. Colour samples of sets S1, S2, S4 and S8 were dyed in light colours (lightness $L^* > 60$), the rest in dark ($L^* < 50$). All studies were conducted in standard climatic conditions [19].

All fabrics were made of yarn of the same linear density, i.e. 100 tex. The sets differed among each other by the fibrous composition of yarn, type of weaving and fabric density. Each set was prepared in two versions, i.e. undyed and dyed. The undyed samples had a natural wool shade. The dyed samples of sets S1, S2, S4 and S8 were made in light shades (lightness $L^* = 60$), and the rest in dark shades ($L^* = 50$). Figure 1 shows an example of a set of textile materials selected for the study.

The dyeing of the fibrous mixture to obtain dyed yarn was carried out according to the technological regime developed by and operating at the Vladi enterprise [18]. At each stage of the processing of textile materials (spinning, weaving and finishing), samples were taken according to the method described in the standard GOST 20566-75. The undyed and dyed samples of yarn and fabric were made on the same equipment, which made it possible to obtain the same experimental conditions.

All experiments were performed under standard climatic conditions [19]. During the study, the structure of samples was not subjected to mechanical deformation and the villi were in the same orientation they acquired during the stabilisation.

2.2 Hairiness measurement

To measure the hairiness of yarn and fabrics, the optical method was used, the essence of which is to determine the amount of hair from sample micrographs [20]. However, the use of this method in the textile industry to assess the indicators of material hairiness has some peculiarities due to the difference in the structure of yarn and fabric. The index of yarn hairiness is thus determined by the total length of fibres protruding on both sides of the body of the yarn [21], and for the fabric, hairiness is determined by the total length of fibres protruding above the fold of one side of the fabric [16]. The indicators obtained in this way do not allow for a comparative analysis of the hairiness of textile materials, which is planned in this work. Therefore, in this study, changes were made to the experimental procedure in the stage of sample preparation. The yarn for the research was previously reeled up on plates of 5 cm × 5 cm in increments equal to the diameter of yarn to obtain a solid covering that simulates the surface of the fabric. The hairiness of both yarns and fabrics was determined at the fold, the contour of which was analysed using a software application [21]. When measuring hairiness, a layer of surface (tangled) pile was taken into account (cf. Figure 2), the boundaries of which were set by the operator based on the definition of

a)

b)

Figure 1: Example of set of samples for research (yarn, raw fabric and raised fabric) in two versions: a) undyed and b) dyed

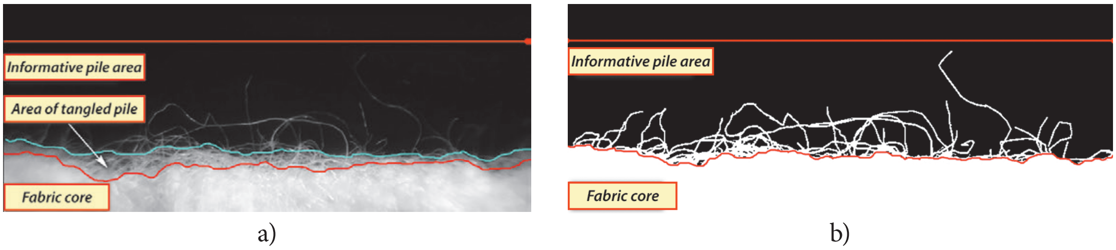


Figure 2: Image of fabric fold with processing zones: a) actual image of fold, b) image in binary format for analysis

the pile layer [8, 22]. The hairiness of the fabric was determined at the fold, which was formed along the warp and weft threads, and along the diagonal. This allowed us to level the influence of the weaving pattern and increase the objectivity of the research results. The amount of pile in the experiment was estimated by the hairiness index H_{tm} of the textile material, which shows the total length of fibres of the yarn (fabric) at the fold of a 1-cm long textile sample. When determining the hairiness of a fabric, the total number of experiments was calculated in a preliminary experiment with a 95% probability used for the textile industry [23]. Ten samples were made from one fabric. For each sample, three experiments were repeated. Taking into account that eight sets of textile materials were examined in two versions (undyed and dyed), the total number of experiments (separately for raw and raised fabrics) was 10 × 3 × 8 × 2 = 480. As a result, the reliability of the obtained results was ensured.

2.3 Colour measurement

The spectral characteristics of yarn and fabric samples were obtained using a system for measuring, evaluating and reproducing colour, consisting of a Spectra Scan 5100 spectrophotometer (Premier Colerscan) and applied programs, which allow solving problems in industrial coloristic applications [24]. The general principles for measuring fabric colour are in accordance with ISO 105-J01: 1997 - Textiles - Tests for colour fastness - Part J01: General principles for measurement of surface colour. This is a current standard version, which was last revised and confirmed in 2018.

The measurement of the colour characteristics of samples and their entry into the software database were conducted under the conditions that ensure the measurement reproducibility of chromaticity parameters. The colour parameters of samples were repeatedly measured using the maximum available viewing area of the spectrophotometer used, i.e. a large aperture (30 mm LAV aperture). The parameters of samples were measured on a backing material similar to a white standard calibration plate (titanium dioxide) used to calibrate the device. The backing material was the same for all samples during the measurement.

To obtain the required accuracy of colour characteristic measurements, each elementary spot sample was positioned on the measuring device, followed by rotation by 10° before carrying out the next measurement. After determining the required number of measurements (measurements with removing the sample from the device with an error not exceeding 0.15 units of chromaticity error ΔE), four control cycles of colour parameter measurements were performed in order to confirm that the averaged result for each of the four cycles was included in the permissible error ΔE = 0.15 units.

In this work, the spectral characteristics of samples were evaluated under standard illumination D65/10, the values of the colour coordinates for which are shown in Table 2.

Table 2: Values of colour coordinates under illumination D65/10

Light source	A	B	C	D_{65}
X_0	109.83	99.07	98.07	95.02
Y_0	100.00	100.00	100.00	100.00
Z_0	35.55	85.22	118.22	108.81

Colour differences were calculated using the CIE L* a* b* (1976) system. Since the essence of the study of colour characteristics was to determine their change depending on the change in the hairiness of textile samples in the process of technological processing, the colour indices of the textile material with the least hairiness were taken as a standard. The following indicators were used to analyse colour characteristics: lightness L*, saturation C* and colour difference dE*. The change in the colour characteristics of samples relative to the reference sample from the corresponding set was analysed by the difference in lightness DL*, difference in saturation DC*, differences in the coordinates Da*, Db*, and the colour difference dE*.

3 Results and discussion

3.1 Changes in colour characteristics of undyed textiles

The results of colour characteristics are demonstrated on the example of the set S2. Figure 3 shows micrographs of textile samples from this set, which were used to determine the hairiness index.

According to the results of the analysis of microphotographs, the hairiness of the yarn had the lowest values; therefore, yarn was chosen as a reference when comparing the colour characteristics of textile samples. The change in hairiness was reflected primarily in the lightness indicators of textile materials. Thus, the lightness of the raised fabric increased in comparison with the yarn, and the colour itself became more yellow. Figure 4 shows the average yarn-to-raised-fabric colour match results from the spectrophotometer analysis using standard test geometry.

Changes in hairiness ΔH_{tm} and colour characteristics of fabrics, presented as a percentage increase relative to yarn (yarn – raw fabric, yarn – raised fabric), and the differences between the corresponding characteristics of raw and raised fabrics are shown in Table 3. Based on the results shown in Table 3, the graphical dependencies of the difference in lightness and colour saturation of undyed textile samples on the changes in their hairiness index are presented in Figure 5.

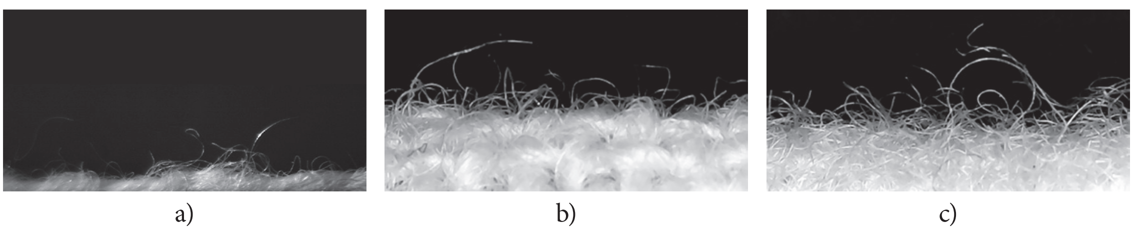


Figure 3: Layer of surface pile on: a) fold of yarn, b) raw fabric, and c) raised fabric

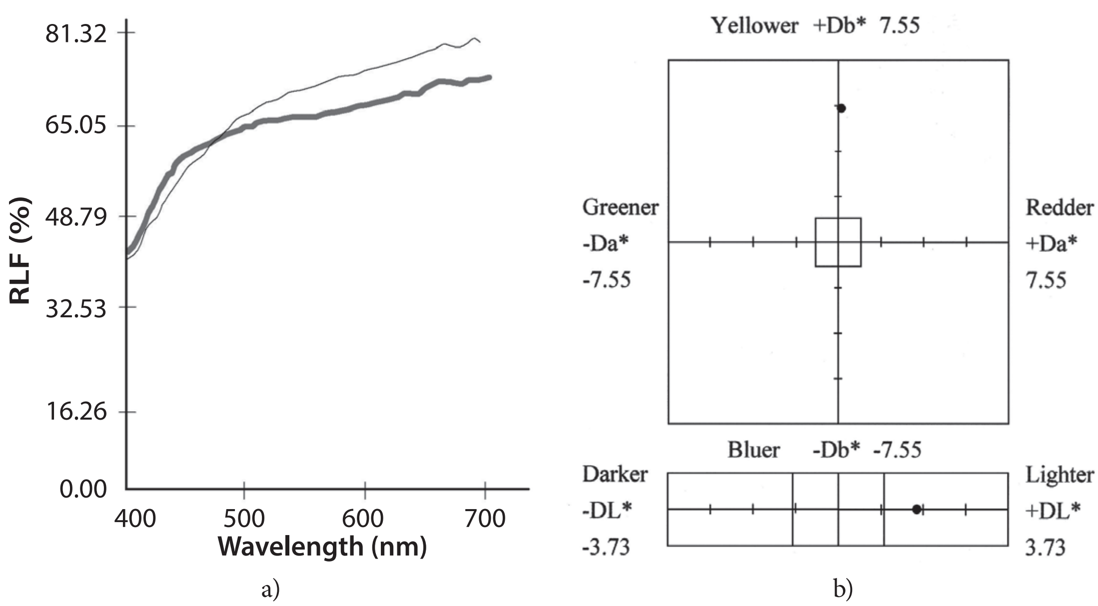
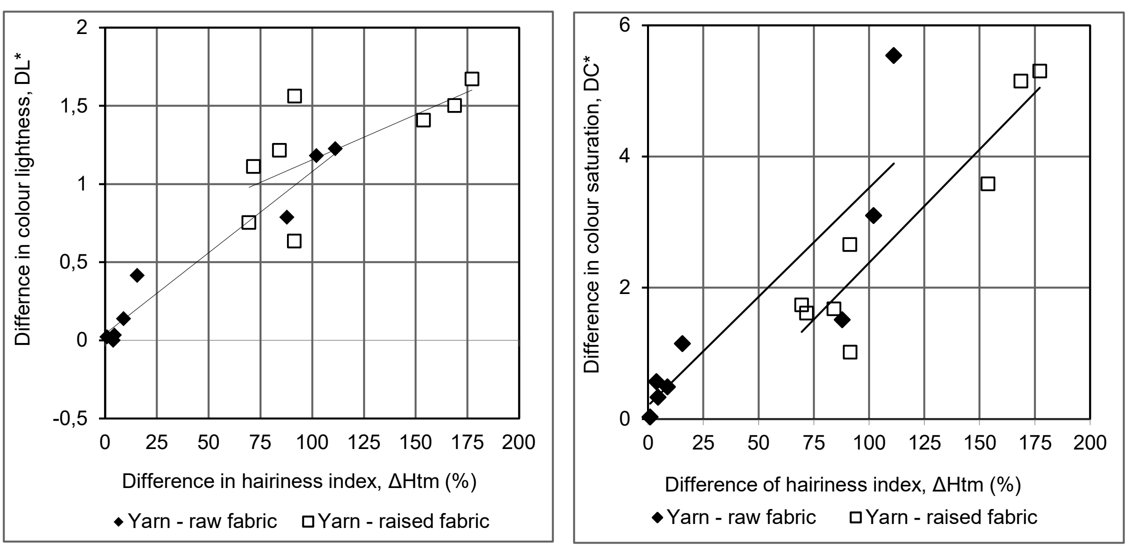


Figure 4: Colour matching of: a) undyed yarn and b) raised fabric from set S2

Table 3: Results of comparison of colour characteristics of undyed textile samples

Yarn – raw fabric								
Textile set	S1	S2	S3	S4	S5	S6	S7	S8
ΔH_{tm} (%)	15.53	102.06	87.82	111.22	4.47	0.91	8.82	3.85
DL*	0.42	1.18	0.79	1.23	0.03	0.02	0.14	0.00
Da*	−0.02	0.55	0.67	0.12	0.03	0.00	0.1	0.06
Db*	1.14	3.01	1.5	5.54	0.35	0.03	0.47	0.56
DC*	1.15	3.10	1.51	5.54	0.33	0.03	0.49	0.57
dE*	1.21	3.28	1.82	5.68	0.35	0.04	0.5	0.57
Yarn – raised fabric								
Textile set	S1	S2	S3	S4	S5	S6	S7	S8
ΔH_{tm} (%)	91.3	168.72	153.78	177.21	84.15	71.69	69.49	91.45
DL*	0.63	1.50	1.41	1.67	1.22	1.11	0.75	1.56
Da*	−0.22	−0.15	0.32	0.34	0.40	−0.25	−0.38	−0.13
Db*	2.62	5.14	3.41	5.3	1.64	1.43	1.99	1.53
DC*	2.66	5.15	3.59	5.31	1.68	1.61	1.74	1.02
dE*	2.71	5.36	3.71	5.57	2.08	1.83	2.16	2.19
Raw fabric – raised fabric								
Textile set	S1	S2	S3	S4	S5	S6	S7	S8
ΔH_{tm} (%)	65.59	89.01	84.87	83.04	70.7	71.09	65.74	75.56
DL*	0.42	1.18	1.67	1.52	1.21	1.36	1.00	0.84
Da*	−0.22	−0.13	−0.03	−0.12	−0.20	−0.08	−0.19	−0.13
Db*	1.17	3.12	2.99	2.85	1.66	1.35	1.22	1.91
DC*	1.15	3.10	2.95	2.75	1.44	1.14	1.46	1.91
dE*	1.26	3.34	3.42	3.23	2.06	1.91	1.59	2.09



a) b)

Figure 5: Change of colour characteristics for undyed samples

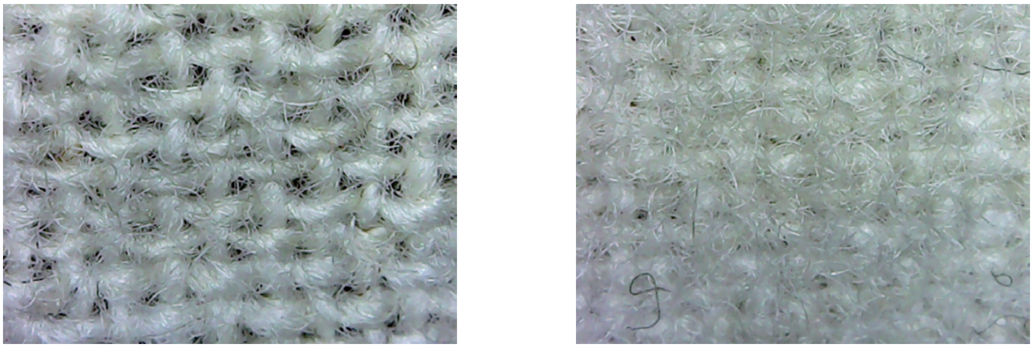


Figure 6: Surfaces of fabric samples from set S2: raw fabric and raised fabric

The analysis of graphic data showed that with an increase in the hairiness of textile materials, their colour characteristics (lightness and colour saturation) increased in direct proportion. The degree of this increase was manifested to a greater extent for the hairiness of the fabrics, since a thick layer of the surface pile covered uneven relief and pores formed by threads (cf. Figure 6). With a further increase in hairiness, the changes in the lightness of fuzzy fabrics reduced, which was confirmed by a decrease in the slope of the graph "yarn – raised fabric". Obviously, this moment came after the villi filled the space between the threads in the weave pattern and completely covered the body of the fabric (cf. Figure 6), forming a layer of solid thick pile, which is typical of fabrics after the raising [16].
The results presented in Table 3 show that hairiness increased the most for the fabric sample sets S2–S4 made from wool with a low proportion of chemical fibres. For these samples, the lightness of the L* colour also increased significantly. For the textiles made with the addition of acrylic fibres, the change in hairiness was less pronounced, and for these samples, the lightness DL* was correspondingly lower.
The analysis of the colour of undyed samples by the change in coordinates a* and b* (deviations towards green-purple or blue-yellow) showed that the colour of some raw fabrics and all raised fabrics with greater hairiness became less red (or greener) (negative Da*). As for the increase in Db*, according to the data obtained, with an increase in the hairiness index, their colour changed towards an increase in yellowness, i.e. the natural shade of woollen fibres from which the yarn was made. This suggests that with an increase in the hairiness of textiles, its natural subtone becomes more noticeable on the surface, which may be the subject of further scientific research.

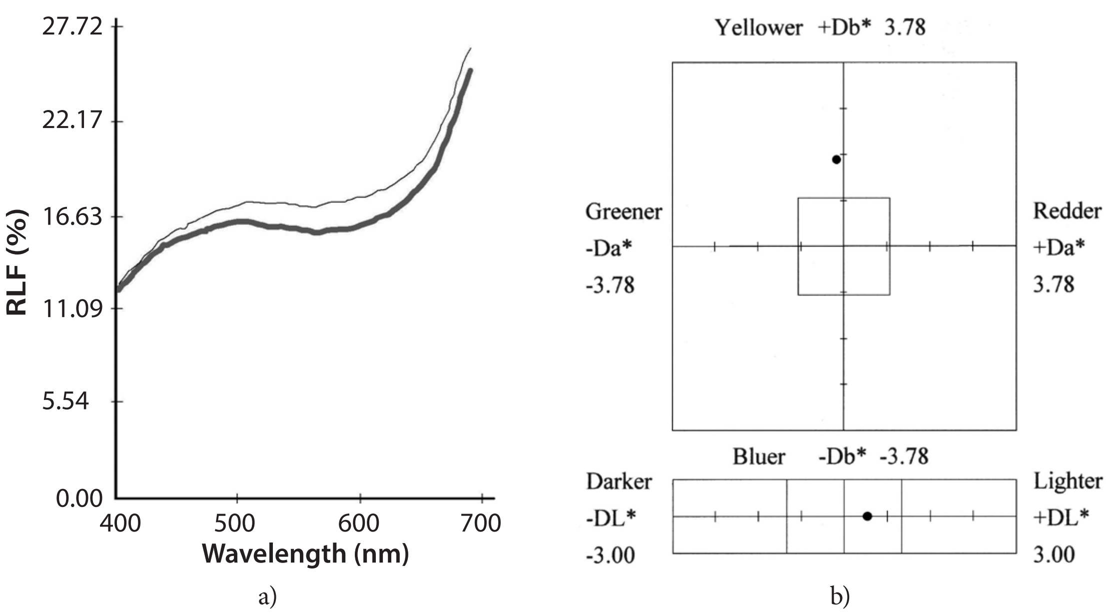


Figure 7: Colour matching of: a) dyed yarn and b) raised fabric from set S3

3.2 Changes in colour characteristics of dyed textiles

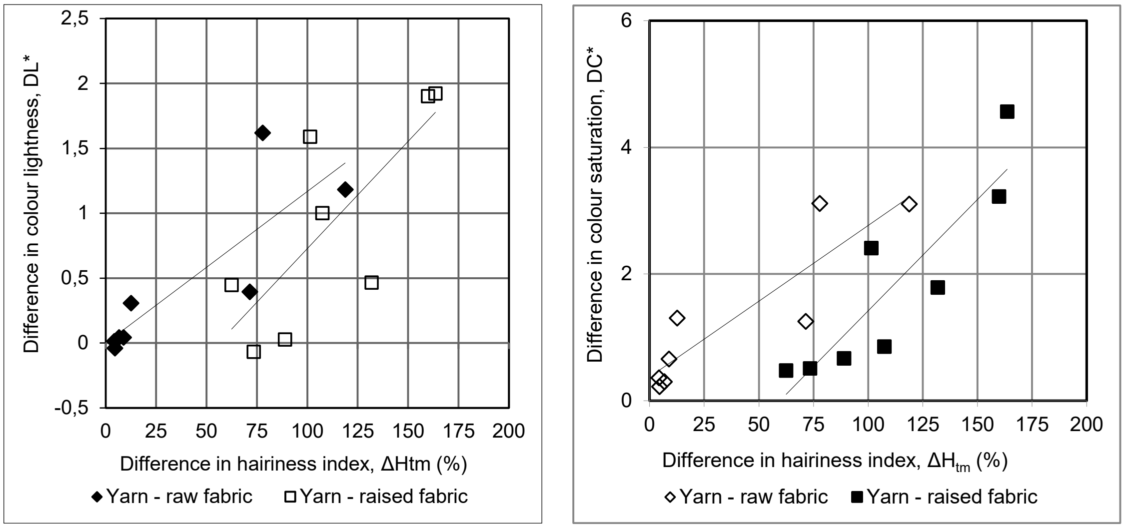
Figure 7 shows the average yarn – raised fabric colour match results from the dyed sample set S3, as measured by a spectrophotometer with standard test geometry.

The changes in hairiness ΔH_{tm} and colour characteristics for dyed textile samples are shown in Table 4. According to the results presented in Table 4, the graphical dependencies of the changes in lightness and colour saturation of dyed fabrics on the changes in their hairiness index were also plotted (cf. Figure 8).

The analysis of graphic data showed that the changes in the colour characteristics of undyed and dyed textile samples from their hairiness index had similar regularity: with an increase in the hairiness index, the values of colour characteristics increased proportionally. However, a certain correction in colour matching was made by the initial value of the lightness of samples, which differed significantly in initial conditions for dyed and undyed textile materials. Therefore, with almost the same increase in the hairiness index for undyed fabrics (cf. Figure 5), the level of change in colour characteristics increased with an increase in hairiness, and for dyed fabrics (cf. Figure 8), the level of lightness and saturation changes with an increase in hairiness did not practically change ("yarn – raised fabric" graphics). It should be assumed that the presence of dark tones in sets of fabrics, which are known to absorb light more than reflect, plays a significant role in the value of lightness of dyed fabrics. The increase in hairiness was reflected to a lesser extent in the level of their colour characteristics. Hence, a study of colour characteristics of dyed fabrics should be performed within groups assembled by colour tone.

Table 4: Results of comparison of colour characteristics of dyed textile samples

Yarn – raw fabric								
Textile set	S1	S2	S3	S4	S5	S6	S7	S8
ΔH_{tm} (%)	12.54	118.8	71.49	77.81	6.69	4.59	8.8	4.27
DL*	0.31	1.18	0.39	1.62	0.04	−0.04	0.04	0.01
Da*	−0.12	0.23	−0.25	0.21	0.39	0.34	0.73	0.60
Db*	1.30	3.1	0.35	3.10	0.3	0.22	0.65	0.35
DC*	1.30	3.10	1.25	3.11	0.3	0.22	0.65	0.36
dE*	1.34	3.32	0.58	3.51	0.49	0.40	0.98	0.7
Yarn – raised fabric								
Textile set	S1	S2	S3	S4	S5	S6	S7	S8
ΔH_{tm} (%)	101.35	159.83	131.82	163.58	62.47	73.39	107.39	88.89
DL*	1.59	1.90	0.47	1.92	0.45	−0.07	1.00	0.03
Da*	−0.1	−0.18	−0.03	−0.34	0.61	0.49	1.2	0.74
Db*	1.17	3.31	1.78	4.45	0.46	0.42	0.57	0.47
DC*	2.41	3.22	1.79	4.56	0.48	0.51	0.85	0.67
dE*	1.97	3.82	1.84	4.86	0.89	0.65	1.66	0.88
Raw fabric – raised fabric								
Textile set	S1	S2	S3	S4	S5	S6	S7	S8
ΔH_{tm} (%)	61.14	82.15	83.37	78.76	50.39	62.46	80.91	65.79
DL*	0.75	1.55	0.43	1.69	0.67	−0.05	0.1	1.34
Da*	0.08	−0.20	−0.14	0.02	0.83	−0.34	2.11	0.1
Db*	1.73	2.32	1.18	2.64	0.44	0.65	0.57	1.63
DC*	1.11	2.64	1.2	2.59	0.33	0.52	2.04	2.00
dE*	1.41	2.74	1.33	2.74	0.88	1.54	2.82	1.72



a) b)

Figure 8: Change of colour characteristics for dyed samples

For dyed textile samples, the difference Da* had both positive and negative values, which also indicates a redistribution of red and green colours, and that the level depends on the initial value of the colour tone. Positive values of Db* indicate that with an increase in the nap layer, the colour tone also deviates towards an increase in yellowness, i.e. a subtone of fibres appears.

Figure 9: Fabric from set S2 before rising (top) and after rising (bottom)

3.3 Colour difference dE*

The difference in the colour characteristics for examined textile samples with different values of hairiness was most clearly demonstrated by the dE* value presented in Tables 3 and 4. For human perception, the dE* value between 0 and 1 indicates that there is no significant difference between the compared colours, and the observer sees the same colour. The slight difference within these limits can only be determined by instrumental methods. The difference between colours with dE* > 1 can be determined by an experienced colourist. To an ordinary observer, the differences between colours become noticeable at dE* ≥ 2. If dE* > 5, the observer sees two different colours [25]. With a hairiness increase of 70% or more, the colour difference between textile materials exceeds 1 and becomes visually noticeable. A particularly significant (more than 2) colour difference was manifested for undyed samples of sets S2, S4 and could be seen with the naked eye (cf. Figure 9). As it can be seen from Figure 9, a visual comparison of two samples of undyed raw and raised fabrics of set S2, for which dE* = 3.34, showed a significant difference in colour indicators, i.e. lightness and saturation of the raised fabric were greater. The same level of colour difference was observed for the dyed sets of textiles S1, S2, S4 (light colour). For the textile samples the lightness L* of which was below 40 (dark tones), an increase in ΔH_{tm} did not cause a colour difference between them. The colour difference of the dyed textile samples with an increase in hairiness was not so noticeable for an average observer, and the deviation from a given shade occurred more often under the influence of a subtone of villi. For example, for the sets of samples S3, S5 and S6, a significant increase in hairiness did not cause significant changes in colour characteristics, while for the undyed samples of the same sets, the colour difference reached 2 or more. For the textiles of dark shades, when the reflection of light rays from a dark fuzzy surface creates the effect of gloss rather than lightness, it is advisable to supplement the study with an analysis of gloss parameters [15].

4 Conclusion

1. Numerous investigations show that hairiness is an important formative factor in the optical properties of textile materials, since it affects not only the technological processes of dyeing textiles, but also the visual perception of the surface of the material and the textile product as a whole.
2. As a result of our experiment, it was found that with an increase in the hairiness of woollen fabrics, their colour characteristics (lightness and colour saturation) increase in direct proportion. However, the degree of increase in lightness and chroma for undyed and dyed samples is different, and depends on the initial colour tone of the fabrics. With almost the same increase in the hairiness index for undyed fabrics, the level of change in colour characteristics increases, while for dyed fabrics, it does not practically change. The study of the colour characteristics of dyed fabrics must be conducted within groups assembled by colour tone.
3. The degree of increase in lightness and saturation is higher for raised fabrics, since a thick layer of surface pile closes relief irregularities and pores formed by threads. However, with a certain amount of hairiness, the increase in lightness decreases and subsequently remains unchanged.
4. The analysis of the colour of tissue samples by changing the coordinate Da* indicates a certain redistribution of constituent colours in the structure of the colour tone, which is not visually determined. Positive values of the Db* coordinate show that with an increase in the layer of hairiness, the colour of fabrics changes in the direction of increasing yellowness (natural shade of wool fibres the yarn is made of), i.e. the natural subtone of fibres becomes more noticeable on the surface of fabrics.
5. Moreover, it was established that the colour mismatch between woollen fabrics with different degrees of hairiness becomes visually noticeable with a total colour difference of $dE^* \geq 1$, which corresponds to an increase in hairiness of 70% or more.
6. The factor of the influence of hairiness on colour effects in textile materials should be taken into account when designing fabrics from dyed yarn, when developing the colour design of fabrics and when calculating technological dyeing modes to ensure the most accurate colour matching to a given sample.

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Acoustic Investigation of Textile Fabrics
Akustična raziskava tekstilnih tkanin

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Abstract
Why is it possible to distinguish between different textile fabrics by just touching them and moving your hand over them and listening to the sound? Particularly for high-quality woven fabrics, e.g. used for tailor-made suits, it is quite common that the dressmaker listens to the sound as their hand rubs the fabric. Can this approach be translated into a technical measurement? What could a sound analysis tell us about the fabric properties? As a first simple approach, we used a record player to rotate different cotton fabrics, and tested fine tips from diverse materials, such as plastic pipettes, pens, glass tips, etc. Our results show clear differences between the textile fabrics, which can be attributed to different yarn, knitted or woven structures. While the rotational mode of investigation impedes fully automated fast Fourier transform (FFT) evaluations, our first results suggest developing this promising method further.
Keywords: sound analysis, woven fabrics, knitted fabrics, hairiness, surface structure, roughness.

Izvleček
Zakaj je mogoče razlikovati med različnimi tekstilnimi tkaninami tako, da samo premikate roko po njej in poslušate zvok? Predvsem za visokokakovostne tkanine, npr. take, ki se uporabljajo za obleke po meri, je zelo pogosto, da krojačica posluša zvok svoje roke, ki se premika po površini blaga. Ali je mogoče ta pristop tehnično izmeriti in kaj bi nam takšna zvočna analiza lahko povedala o lastnostih tkanine? Kot prvi preprost pristop smo s pomočjo predvajalnika gramofonskih plošč vrteli različne bombažne tkanine in preizkušali fine konice iz različnih materialov, kot so plastične pipete, pisala, steklene konice itd. Naši rezultati kažejo jasne razlike med tkaninami, ki jih lahko pripišemo različnim prejam, pletenim ali tkanim strukturam. Medtem ko rotacijski način preiskave ne omogoča popolnoma samodejne ocene hitre Fourierjeve transformacije (FFT), so naši prvi rezultati spodbudni za nadaljnji razvoj te obetavne metode.
Ključne besede: analiza zvoka, tkanine, pletiva, kosmatost, površinska struktura, hrapavost

1 Introduction

Textile fabrics are often used for sound absorption or acoustic insulation and investigated for this purpose by many research groups [1–5]. Some groups also work on developing acoustic sensors embedded in yarns or fabrics [6].
Only recently has the issue been addressed whether acoustic investigations of textile fabrics, i.e. performing an analysis of the sound created by frictional movement along their surfaces [7, 8], could reveal information about the fabrics. Wang *et al.* studied the friction sounds created by woven fabrics from natural fibres rubbing on each other and found frequency-dependent loudness, averaging over time [9]. Loudness was also the parameter investigated in some other studies, e.g. by Yi and Cho, examining different woven fabrics [10, 11]. Afterwards,

Yi *et al.* as well as Kim *et al.* added subjective pitch, clearness and sharpness to the loudness, and found correlations with different mechanical properties, as measured with a Kawabata KES-FB [12, 13]. The same sound generator, rubbing two fabrics under a defined load against each other, was also used to investigate warp-knitted and weft-knitted fabrics in terms of loudness and loudness range [14, 15]. A comprehensive study on knitted and woven fabrics with a focus on water repellent, vapor permeable fabrics was carried out by Park and Cho, comparing acoustic and mechanical parameters [16].

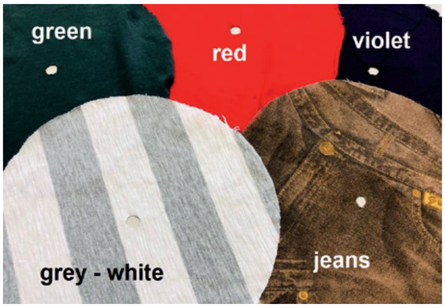
A slightly different approach was chosen by Yosouf *et al.* [17]. They developed an instrument which follows the trajectory of a moving arm along the side of the body, in this way for the first time not following a linear trajectory, i.e. moving two fabrics against each other not exactly along warp or weft direction, but along a bent line. Evaluation of the acoustic measurements concentrated on the amplitude, as in most previous studies.

Here, another approach is chosen. Instead of averaging over large areas by investigating textile-textile friction, we perform an acoustic analysis using fine tips from different materials, enabling time- and spatially resolved sound examination. In this way, it is generally possible to acoustically count warp and weft densities in woven fabrics and similar numbers in warp and weft knitted fabrics.

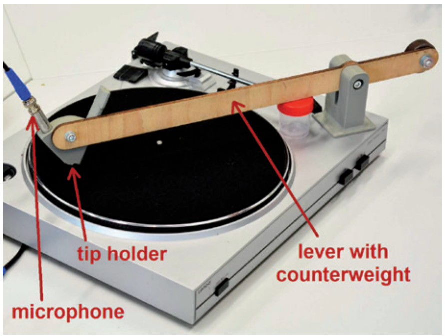
2 Materials and methods

All textile fabrics under examination in this study consisted of 100% cotton. Table 1 gives an overview of the fabric parameters.

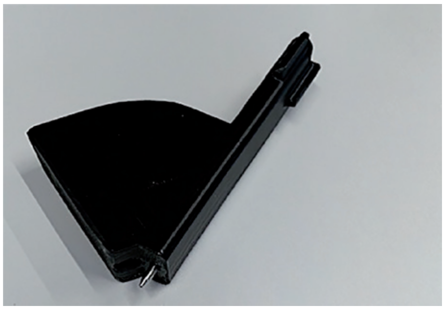
An L3865 record player (Lenco, Nuth, Netherlands) was used to rotate the samples cut to fit the dimension of the turntable. A 130D20 microphone (PCB Piezotronics) attached to an SN 23697 AD-converter



a)



b)



c)

Figure 1: a) Cut samples under examination; b) Measurement setup with the tip holder and inserted microphone (attached to the blue connector) mounted on a lever, which allows it to carefully touch the rotating textile fabric; c) Ballpoint pen refill placed in the tip holder

Table 1: Fabric parameters of the samples under investigation

Name	Structure	Thickness (mm)	Areal weight (g/m^2)	Courses/cm Ends/cm	Wales/cm Picks/cm
Green	weft knitted	0.80	181	18	13
Red	weft knitted	0.59	155	19	15
Violet	weft knitted	1.23	255	8	7
Grey-white	weft knitted	0.81	204	19	12
Jeans	woven	0.79	162	13	15

(National Instruments) was mounted on the record player instead of the common arm in a 3D printed holder for different tips, as depicted in Figure 1, in a distance of 105 mm from the middle of the turntable. The lever with the counterweight (Figure 1b) lets the tip softly touch the fabric (distance of 0 mm) so that the friction does not move the fabric, but a constant connection is ensured. The output voltage signals of the microphone were digitalised by the AD converter (sample frequency of 51.2 kHz, 24 bits) and saved and evaluated by a self-written Matlab code. Generally, the sound is produced by friction between the textile fabric and tip.

Different pipettes and tubes from glass and plastics and a ballpoint pen refill (Figure 1c) were tested as tips. The plastic pipettes were found not to be stiff enough; their own bending caused undesired noise, which is why they were excluded after the first tests. The glass tubes, on the other hand, were not fine enough and impacted the textile fabrics too strongly. Finally, a stainless-steel ballpoint pen refill without dye (no-name, refill diameter of 2.51 mm, ball diameter of 1.5 mm) was chosen for the main tests.

3 Results and discussion

Firstly, Figure 2 depicts exemplary time-domain results of measurements on the "red" sample (Figure 1a) on different time scales. Generally, the record player was set to 33/min, i.e. 0.55 Hz, meaning that one rotation takes 1.82 s (denoted by vertical black lines). Each measurement was performed during 6 full rotations, so that the values in the middle of the measurement duration are free from any possible bias due to starting and ending the rotational movement.

Starting with the complete measurement along 6 rotation cycles (Figure 2a), there are no regularities clearly visible. Examining exactly one rotation cycle (Figure 2b) results in approx. 5–7 minima

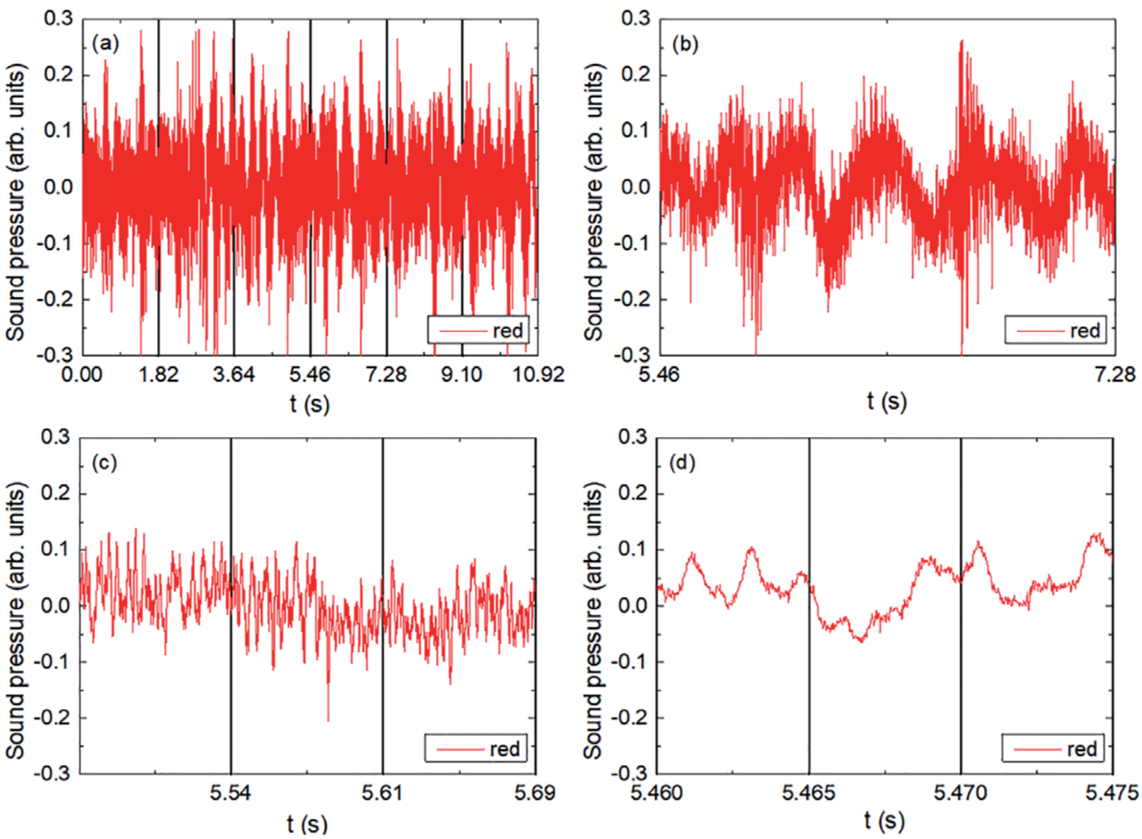


Figure 2: Measurements of time-dependent sound pressure of the red sample for different time scales: a) full measurement over 6 cycles, b) measurement during the 3rd cycle, c) measurement during the first 45° of the 3rd cycle, and d) measurement during the first 3° of the 3rd cycle

and maxima, depending on how exactly these are counted, with varying amplitudes and wavelengths. Generally, a fourfold anisotropy may be expected due to the symmetry of a knitted fabric, which is, however, not visible here.

Next, Figure 2c further decreases the time section under investigation, this time to an angle of 45° between the first and last depicted results. Now, the short-time variations inside the longer ones, seen in Figure 2b, also become visible. Finally, the time is reduced to ~ 0.015 s, corresponding to an angle of 3° which is depicted in Figure 2d. Here, the short-term variations become well visible, together with even shorter variations that can be attributed to noise.

These oscillations are still not harmonic, which obviously results from the steady rotation of the system, as opposed to a linear movement, in which threads should be passed in continuous durations.

Next, Figure 3 depicts sections of 3° (corresponding to Figure 2d) of the other four fabrics. Here, we see clear differences, especially between the knitted and the woven fabric (“jeans”), while the coarsest knitted fabric (“violet”) does not show such strong deviations from the others.

To understand these differences, it is supportive to compare the average numbers of yarns per centimetre with the measured frequencies. This is done here for the jeans woven fabric.

According to Table 1, this fabric has an average number of 14 yarns/cm, assuming that counting occurs along the orientation of the warp or weft yarns, which is not the case here. For a rough estimate, however, we can assume that the well-visible maxima and minima belong to crossing one yarn at a time, while some lateral sliding correlated with shifting a yarn may occur in the more noisy regions, e.g. in the left part of Figure 3d. From the radius of the circle, on which the ballpoint pen refill is moved (105 mm), a circumference of 660 mm can be calculated, which is measured within 1.82 s. For the areas, in which measurement takes place more or less parallel to the warp or weft yarns, we can calculate a frequency of approximately 508 yarns/s, i.e. an expected time distances between subsequent maxima of approximately 2 ms.

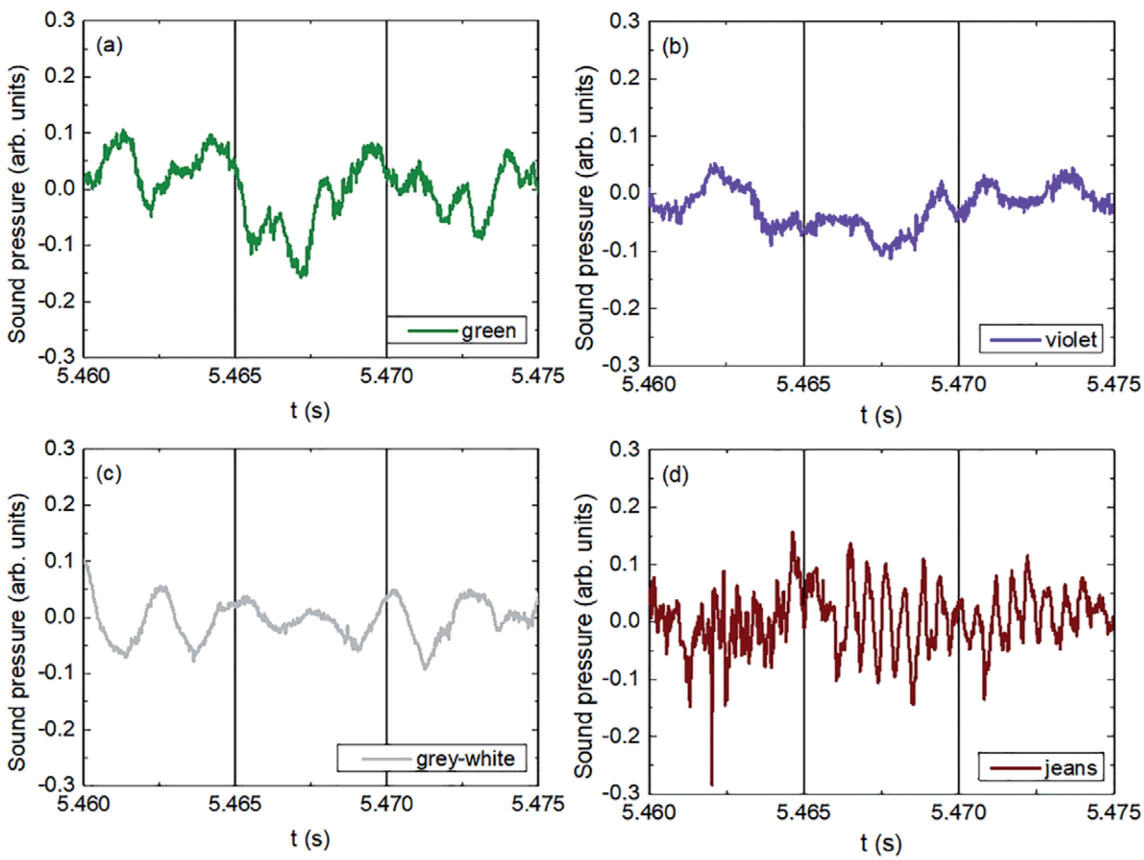


Figure 3: Measurements of time-dependent sound pressure of the other samples for identical durations: a) green, b) violet, c) grey-white, and d) jeans fabrics

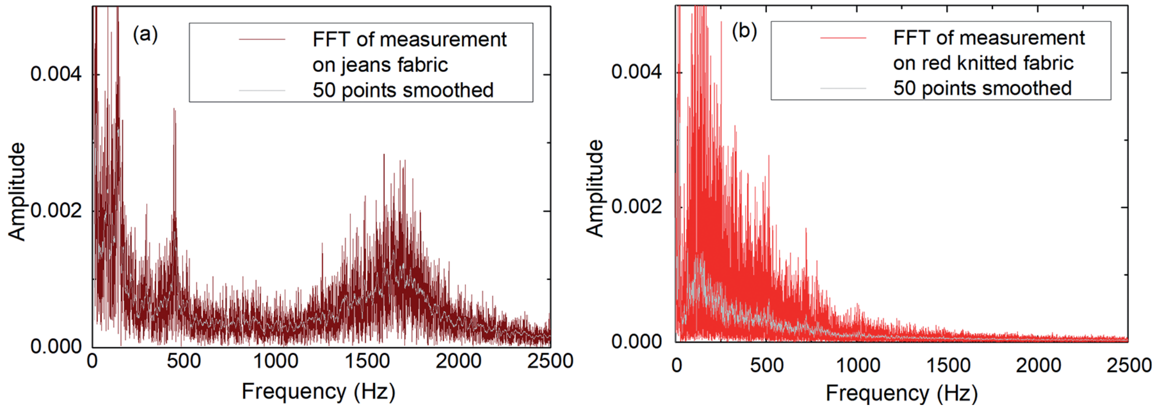


Figure 4: FFT spectrum of the full measurement on the jeans fabric (a) and the red knitted fabric (b)

For the right part of Figure 3d, a much smaller value of approximately 0.59 ms is calculated (i.e. ~ 1690 Hz), while in the left part as well as in other parts of the whole graph (not shown here), another time distance can be detected, i.e. approximately 2.1 ms (470 Hz). These values indicate that in general, the clear oscillations are correlated with the ballpoint pen refill crossing the yarns since both occur on the same time scale. However, a more sophisticated calculation is necessary to evaluate the warp and weft densities acoustically.

It must also be mentioned that the highly irregular oscillations of the knitted fabrics suggest finding better methods than manual estimations of the average wavelength, which would be a necessary prerequisite of correlating frequencies to structures. For this, typically a fast Fourier transform (FFT) is applied. Figure 4 clearly shows the FFT spectrum of the measurements on the jeans fabric and on the red knitted fabric, as originally calculated for the whole measurement duration of 10.9 s (6 rotation cycles) and smoothed over 50 points to suppress the noise.

Two main frequencies are visible for the jeans fabric (Figure 4a): approximately 450 Hz and approximately 1650 Hz, both quite near to the manually calculated frequencies for the jeans fabric. The smaller frequency, where a thinner and higher peak is visible, is apparently correlated to the ballpoint pen refill crossing a yarn, as calculated above, while the other peak is much broader and must thus be correlated with the ballpoint pen refill crossing yarns under other angles than 90°, resulting in crossing warp and weft yarns alternatingly and thus leading to higher frequencies. However, as mentioned above, a more sophisticated geometric model is necessary to fully understand all information available from such acoustic measurements.

The FFT of the measurement on the red knitted fabric (Figure 4b), however, shows only one clear peak around 150 Hz and some smaller peaks around 520 Hz, 720 Hz and 1010 Hz. According to the above explained calculation, we would expect for this knitted fabric frequencies of approximately 690 Hz and 1090 Hz (if both yarns in a stitch are crossed) or 540 Hz (if a stitch is crossed near the head where only one yarn is detected), respectively. These expected frequencies fit to the small peaks in Figure 4b. Nevertheless, these peaks are not highly reliable due to the large background. Besides, the larger peak near 150 Hz remains unexplained.

An important factor to evaluate the value of such measurements is the reproducibility. It was addressed with measurements during 6 full rotation cycles. Figure 5 depicts results of subsequent measurements on the woven jeans fabric and on one of the knitted fabrics. A time was chosen where the woven fabric showed a change in the oscillation amplitude to enable comparison between subsequent rotation cycles. For the woven fabric (Figure 5a), the first curve is clearly lower and varying stronger due to the starting process of the measurement. The others all show a reduced amplitude in a similar range, spaced by one rotation (marked by the black arrow), and also other similarities. It should, however, be mentioned that textile fabrics are soft and not completely inelastic (even a woven fabric) and it is thus obvious that – opposite to a metal plate or similar – the positions measured will always vary slightly due to the interaction between the measurement tip and fabric. The measured frequencies, however, should be similar. FFT values for the short durations depicted here allow for only a rough estimation; nevertheless, the values depicted in Figure 5a are mostly similar, except the first rotation.

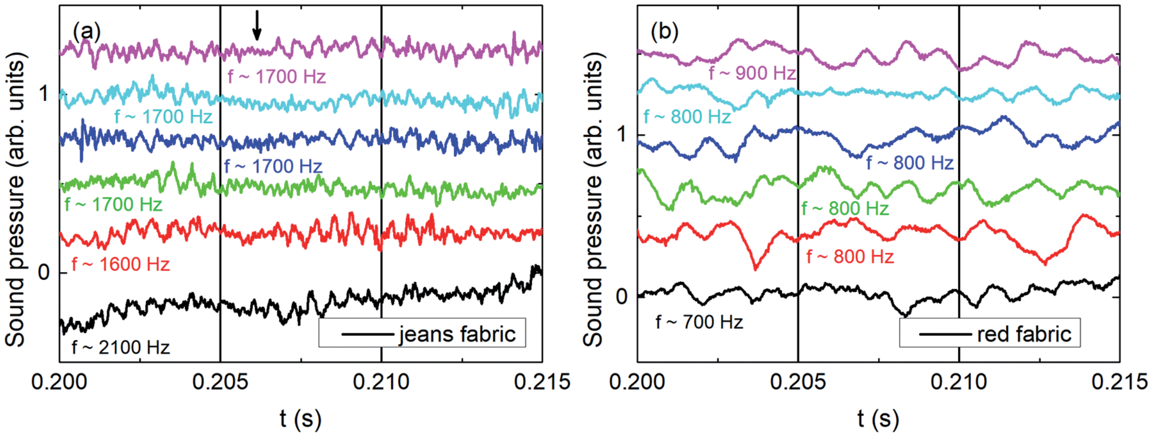


Figure 5: Time-dependent sound pressure measurements at identical positions for (black) first, (red) second, (green) third, (blue) fourth, (cyan) fifth and (magenta) sixth rotation. The lines are vertically offset for clarity by a constant value. Insets depict approximate frequencies, as calculated by FFT for the visible durations. Values are shown for jeans fabric (a) and for red knitted fabric (b).

The results for knitted fabrics are different. On the one hand, frequency measurements by FFT are even less exact here, while the measured frequencies are again similar. It is not possible, however, to find corresponding features in the measurements at the same position during different rotation cycles. Apparently, even a small pressure of the ballpoint pen refill is sufficient to move single threads in the stitches or elongate the knitted fabric so that in each rotation cycle a slightly different position of the knitted fabric is examined. We assume that these problems, which were already recognised in Figure 4b, can be reduced if in the next version the tip is made flexible, e.g. in the form of an elastic metal strip with a fine, rounded tip fixed in the tip holder. Probably, similar to atomic force microscopy (AFM) where the cantilevers have to be chosen according to the samples under investigation and according to the exact questions to be answered, it is necessary to define different tips for samples of different surface roughness, yarn stiffness, hairiness, etc.

4 Conclusion

Acoustic measurements of different knitted and woven fabrics were performed rotating the textile fabrics on the turntable of a record player and measuring the sound of a ballpoint pen refill by a microphone. Time-domain and frequency-domain evaluations allow detecting frequencies for woven fabrics, which can be correlated with the yarn density. On the other hand, more information is contained in the FFT spectra that has to be evaluated carefully. For knitted fabrics, the soft yarns seem to be moved strongly by the ballpoint pen refill, resulting in highly irregular signals due to shifted yarns, which are crossed at random positions within a certain interval, which necessitate a more sophisticated experimental and/or theoretical approach.

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The Effect of Humidified Air on Yarn Properties in a Jet-Ring Spinning System

Vpliv navlaženega zraka na lastnosti preje, izdelane v curkovnem prstanskem predilnem sistemu

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Abstract

In this study, the effect of 100% atmospheric relative humidity on yarn properties was investigated using jet-ring nozzles and compared with the yarn properties of yarns produced with air operated jet-ring nozzles under normal conditions. As a humidification system, a pneumatic conditioner, also known as a lubricant, was used in pneumatic systems. This conditioner was connected just before the pneumatic distributor that supplies air to the nozzles. The tube in stage 2 of the conditioner was filled with pure water at room temperature (25 °C ± 2 °C). The air conditioner dose was adjusted to 100% atmospheric relative humidity. The use of humidified air to jet-ring nozzles had a slight positive effect on all yarn properties (yarn hairiness, yarn irregularity, yarn elongation and yarn tenacity). According to the results, it resulted in a 1% to 3% improvement in yarn quality. This study is the first example and an original study in this field, as there is no study using humidified air in existing jet-ring air nozzle studies. It was proven in this study that humidified air results in a slight improvement in yarn properties.
Keywords: yarn quality, yarn hairiness, jet-ring, nozzle-ring, air nozzle, yarn properties

Izvleček

V tej študiji je bil proučevan vpliv 100-odstotne relativne zračne vlažnosti na lastnosti preje, izdelane z uporabo curkovnih šob, in njena primerjava z lastnostmi preje, izdelane z uporabo curkovnih šob v normalnih okoliščinah. Kot sistem za vlaženje se v pnevmatskih sistemih uporablja pnevmatski vlažilec, ki se uporablja tudi kot mastilna naprava. Ta naprava je nameščena tik pred pnevmatski razdelilnik, ki v šobe dovaja zrak. Cevka mastilne naprave je bila v drugi fazi napolnjena s čisto vodo sobne temperature (25 °C ± 2 °C). Delovanje klimatske naprave je bilo naravnano na 100-odstotno relativno vlažnost zraka. Uporaba navlaženega zraka na curkovni šobi je nekoliko izboljšala vse lastnosti preje (kosmatost, neenakomernost, raztezek in trdnost preje). Rezultati zagotavljajo 1–3-odstotno izboljšanje kakovosti preje. Ta študija je prva na tem področju, saj v obstoječih študijah prstanskih curkovnih šob uporaba navlaženega zraka ni znana.
Ključne besede: kakovost preje, kosmatost preje, curek-prstan, obroč šobe, zračna šoba, lastnosti preje

1 Introduction

Within the textile sector, the measurement and evaluation of yarn hairiness and hairiness variation on the yarn gradient line is an important part of the total quality control of a yarn. Particularly as the result of various technological developments, an increase in machine speeds and an increase in quality expectations, yarn hairiness has become an important yarn parameter that should be controlled through measurement.

Spinning systems used in yarn production function according to the principle of real or false twist. The most typical example of a spinning machine functioning according to the actual bending principle is the conventional ring spinning system. The conventional ring spinning system, one of the first developed spinning systems, has been one of the most widely used spinning methods from the past to the present. One end of a fibre bundle or group of filaments is held constant while the twist type based on the principle of turning the other end along its axis is called a true twist. In contrast, a false twist imparts a temporary twist to the fibre bundle. However, after separation from the twist element, the fibres are parallel and untwisted. As a result, there is no twist on the fibre bundle and therefore no real strength is imparted to the yarn. This principle is contrary to normal yarn with the specific requirement to gain strength. Nevertheless, if the system is modified, it is possible to spin a yarn with a suitable strength value with the false twist principle in the same system.

The most important developments in the field of spinning have been in the field of false twist or winding spinning. The original idea of the false twist principle is based on the addition of fibres to the false twist structure following the removal of the torque at the output of the false twist element and the rotation of the inserted fibres in the opposite direction. This is very similar to the idea that the fibres emerging from the yarn surface are wound around the centre of the yarn. Such fibres trapped in the structure ensure the high cohesion of the yarn even after twisting. Various researchers have used devices and processes that have similar effects on their patents. One of the most important patents is Murata's air-jet spinning system.

The first design for the concept of obtaining fibres by collecting and twisting fibres using a rotating fluid was developed by Götzfried [1]. Götzfried [1] and then Pacholski et al. [2] showed that air jets entering tangentially into the nozzle hole caused a vortex in the nozzle, and could be twisted to the yarn passing through the centre of the rotating air stream at high speeds [3]. The development of modified spinning systems with the addition of air nozzles to various spinning systems and research on the effect of these systems on yarn properties have been studied in recent years.

1.1 Jet-ring (nozzle-ring) spinning system

This system is based on the placement of air nozzles used in the air-jet spinning system between the output system of the conventional ring spinning system and the yarn guide system, referred to as a jet-ring or nozzle-ring (Figure 1). Air is fed into the air nozzle used in the jet-ring system at a certain pressure value. Compressed air creates a rotating air vortex in the nozzle. The air vortex ensures that the fibre ends that protrude outward from the yarn body are wound up in the yarn body, thereby reducing yarn hairiness [5]. In addition to low yarn hairiness, fabrics made from the aforementioned yarns are smoother and more resistant to pilling than fabrics made from conventional ring-spun yarns. Air nozzles thus provide improved yarn properties and long-term advantages due to improvements in fabric performance.

Figure 1: Use of an air nozzle on a jet-ring spinning system

The first experiments with jet -ring or nozzle-ring spinning system were carried out towards the end of the 1990s by Wang et al. [6]. Recently, however, many researchers have been working on yarn properties, in particular on the improvement of yarn hairiness over the use of air nozzles in a conventional ring spinning system [5–9]. Yilmaz [7] evaluated the effect of a pseudo-twist on the yarn properties of compressed air fed into the air ring in the conventional ring spinning system. In order to determine the performance of the core, the yarns produced as Ne 30/1 yarn represent the linear density. In this study, the air nozzle in the conventional ring spinning systems were assembled and produced using yarns, referred in literature as "jet-ring yarn". Yilmaz determined that there was not a single type of flat hair with the lowest hairiness values. It has been determined that the rate of improvement in hairiness values changes depending on the structural parameters and air pressure value. However, it was found that different yarn types were effective on other yarn properties that determine the yarn quality, as well as yarn hairiness. It was determined that a jet-ring modified yarn spinning system consisting of an air nozzle results in a reduction in the number of short fibres of measuring 1 mm and 2 mm, as well as the long hair count.

2 Material and methods

2.1 Air nozzle

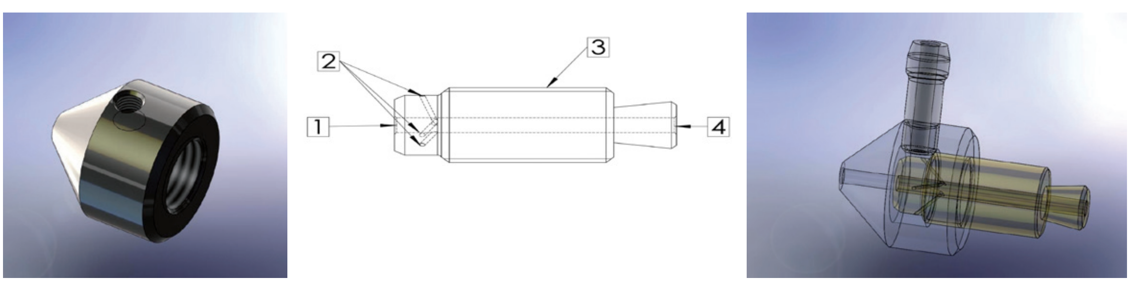
A jet-ring (nozzle-ring) spinning system comprises three basic components: compressed air, nozzle and yarn. Compressed air with a certain value from the compressor is transported to the level and passed through the thread. The nozzle assembly (Figure 2c) has a very simple structure and consists of a nozzle housing (Figure 2a) and nozzle body (Figure 2b). The nozzle body part has a circular cross-section consisting of the twisting chamber (main hole) (1), injectors (2), connecting screw for the nozzle housing (3) and the nozzle outlet (4) (Figure 2b). The main hole extends from the nozzle inlet to the nozzle outlet. The injectors are positioned tangentially to the twisting chamber. The nozzle housing conveys the compressed air from the compressor to the twisting chamber section of the device via the injectors [8, 9]. Flow volume is illustrated in Figure 3. As shown in Figure 3, the design parameters of the air nozzles are composed of twist chamber diameter (*Dtc*), injector diameter (*Di*) and injector angle (θ). A nozzle length is 27mm and twisting chamber diameter of $\phi = 2$ mm was maintained in all samples.

2.2 Experimental setup

The structural parameters of the jet-ring nozzle used to determine the effect on air humidified nozzles are given in Table 1. Ten nozzle types were defined for this experiment. Generally, air nozzles do not work on a bending chamber diameter of more than $\phi = 3$ mm and an injector diameter of more than $\phi = 0.9$ mm [5, 7, 9]. In this study, samples with large twist chamber and injector diameters were preferred in order to minimize the effect of humidified and increased air density on the pressure loss problem. Yarn production was performed using a Merlin SP43 conventional ring spinning machine made by the Pinter Group, with a capacity of 16 spindles. Jet-ring air nozzles mounted on a conventional ring spinning machine are shown in Figure 4.

2.3 Air humidifier system

A pneumatic conditioner, also known as a lubricant, was used as the air humidification system, which is generally connected to the compressor outlet in pneumatic systems (Figure 5). This conditioner was connected just before the pneumatic distributor that supplies air to the nozzles. A typical pneumatic lubricant consists of two stages. In the first stage, the



a) b) c)

Figure 2: Nozzle (a) housing, (b) body and (c) assembly

moisture in the air is maintained, i.e. atmospheric humidity is established. Water is an undesirable feature in pistons, cylinders, pneumatic actuators or similar systems that generally operate within a pneumatic system. For this reason, special pneumatic dryers are also used in large industrial plants. In the second stage of the conditioner, the dried air was lubricated using oil contained in the conditioner tube at the desired dose. The aim of this approach is to ensure that pneumatic systems function with oil, silently and over a long useful life. In this study, it was used outside the purpose of the conditioner. The tube in the second stage of the conditioner was filled with pure water at room temperature (25 °C ± 2 °C) instead of oil. The air leaving the conditioner in this way can be air containing moisture or even water instead of oily air. The aim of this study was not to contain water, but to obtain air with 100% relative humidity. Therefore, before starting the experiment, the conditioner dose was adjusted to 100% relative humidity. Humidity measurements were confirmed using an E+E brand model EE160 temperature and relative humidity transmitter (Figure 6). The EE160 temperature and relative humidity transmitter can

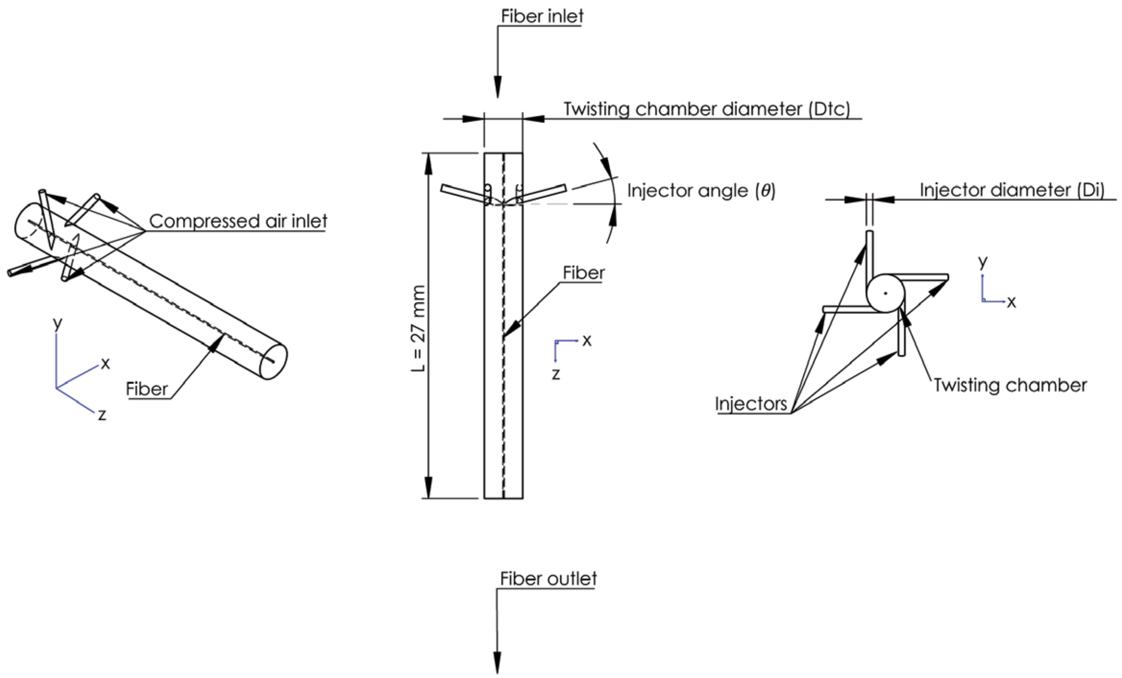


Figure 3: 3D model drawing of a conventional nozzle flow volume with four injectors

Table 1: The structural parameters of the jet-ring nozzle

Nozzle no.	(mm)	Injector diameter (mm)	Injector qty	Injector angle (°)
1	3	0.8	4	20
2	3	0.8	4	25
3	3	0.8	4	30
4	3	0.8	4	35
5	3	0.8	4	40
6	3	0.9	4	20
7	3	0.9	4	25
8	3	0.9	4	30
9	3	0.9	4	35
10	3	0.9	4	40

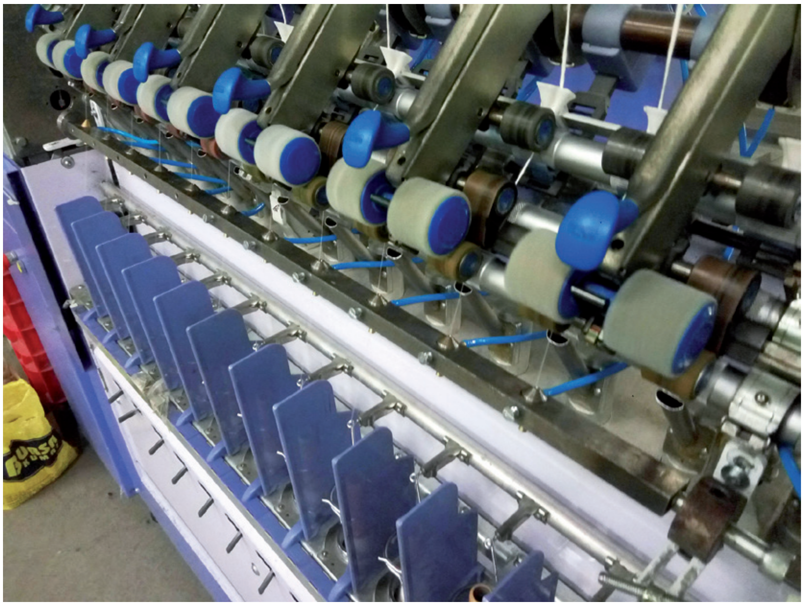


Figure 4: Jet-ring air nozzles mounted on a conventional ring spinning machine

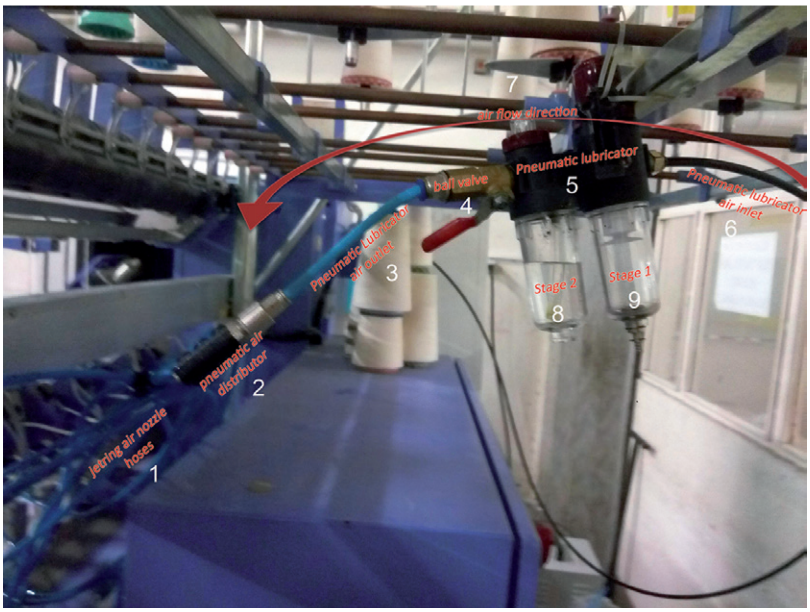


Figure 5: Pneumatic lubricator for use with an air-nozzle humidifier
1 – jetring air nozzle hoses, 2 – pneumatic air distributor, 3 – pneumatic lubricator - air outlet, 4 – ball valve, 5 – pneumatic lubricator, 6 – pneumatic lubricator – air inlet, 7 – air flow direction, 8 – stage 2, 9 – stage 1

be used to measure ambient temperature with an accuracy of ± 0.3 °C and relative humidity with an accuracy of ± 2.5%. With its analog/modbus output, the EE160 can transfer digital data to PLC (programmable logic controller) or HMI (human machine interface, touch screen) systems. In this study, EE160 temperature and humidity transmitter was connected to an electrical panel containing an HMI, and data analysis was performed (Figure 7).

2.4 Yarn production

One-hundred percent cotton yarns were produced in this experiment. We produced cotton jet-ring yarns of 19.6 tex (Table 2). In all yarn productions, importance was given to working with the same spinning parameters, e.g., the same twist multiplier, draft, spindle speed and traveller type (Table 3). The number of injectors was kept constant as four pieces. In all jet-ring yarn productions, the air pressure was kept at 125 kPa (gauge).

Figure 6: E+E EE160 temperature and humidity transmitter

Figure 7: Electrical panel containing HMI

Table 2: Fibre properties

Fibre properties	19.6 tex
Staple length (mm)	30.53
Micronaire	4.52
U.I. (uniformity index)	85.7
Strength (cN/tex)	34.5
Breaking elongation (%)	6.4
SFI (short fibre index)	6.9
+b (yellowness)	7.9
Rd (reflectance degree)	72.1
CG (colour grade)	41–2
SCI (spinning count index)	160

Table 3: Spinning particulars

Parameters	19.6 tex
Roving count (tex)	472
Twist (1/m)	830
α_e	3.7
Mean spindle speed (rpm)	13000
Take up speed (m/min)	15.7
Traveller type	SFB 2.8 PM udr
Traveller ISO No.	31.5–50
Ring diameter (mm)	38
Draft/Break draft	1.181
Total draft	50.4

2.5 Yarn tests

Yarn hairiness, irregularity and imperfection tests were carried out using an Uster Tester 3. Tensile property (percentage of elongation and tenacity measured as cN/tex) tests were carried out using an Uster Tensorapid. The cops and bobbins of each system were fed in the same order to the testers. Yarn test details are given in Table 4. The tests were

Table 4: Test particulars for each yarn sample

Yarn properties	Test device	Test length (m)	Test number	Total length (m)
Yarn irregularity and imperfections	Uster Tester 3	400	1	400
Yarn hairiness	Uster Tester 3	400	1	400
Tensile properties	Uster Tensorapid	0.5	10	5

carried out under the same atmospheric conditions (75% ± 5% relative humidity and 25 °C ± 2 °C), and we conditioned samples for a minimum of 72 hours before the tests. All the tests were carried out on the same testers and test results were analysed statistically to determine any significant differences.

3 Results and discussion

3.1 Yarn hairiness results

The effect of conditioned and normal air on yarn hairiness (H) is illustrated in Figure 8 for an injector diameter of ϕ = 0.8 mm and in Figure 9 for an injector diameter of ϕ = 0.9 mm. In both injector diameter values, normal air and conditioned air resulted in similar hairiness values in the first measurement. In the second measurement one week later, an increase in hairiness was expected, as the yarn was completely dry. On the contrary, however, a surprising slight reduction in hairiness occurred. For yarn produced using a humidified air nozzle, hairiness values in the second measurement (after one week) decreased by approximately 0.1–0.2 compared to the hairiness values in other measurements. Conditioning (humidified) air improves the yarn hairiness value. Expressed in percentages, it means an improvement in hairiness of about 1.5% to 3.5%.

3.2 Yarn irregularity results

The effect of conditioned and normal air on yarn irregularity (% Cv m) is illustrated in Figure 10 for an injector diameter of ϕ = 0.8 mm and in Figure 11 for an injector diameter of ϕ = 0.9 mm. A slight decrease in yarn unevenness was observed according to the condensed air measurements of an injector diameter of ϕ = 0.9 mm. The situation is slightly different for condensed air measurements of yarns produced in configurations with an injector diameter of ϕ = 0.8 mm. A reduction in yarn irregularity was observed when using humidified air in 20° and 25° nozzle configurations. However, the yarn irregularity value increased in both in a 30° nozzle configuration, and in a 35° nozzle configuration (second condition) and in a 40° nozzle configuration (first condition). Nevertheless, in general, it can be said that the yarn irregularity value is reduced by 0.1% to 0.4% in yarns produced with humidified air. Humidified air was observed to improve both yarn irregularity and yarn hairiness.

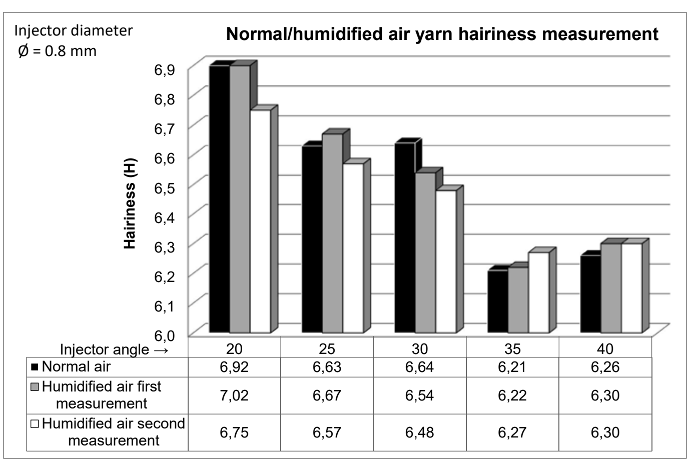
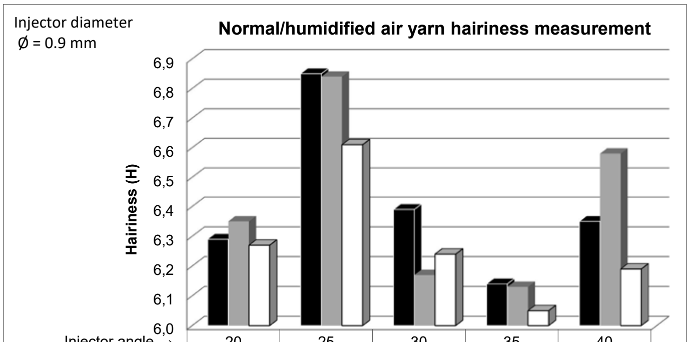
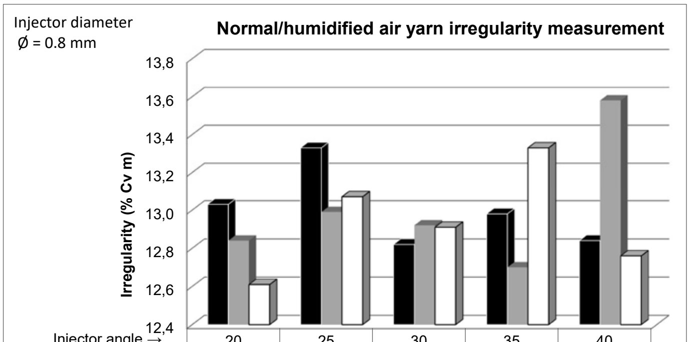


Figure 8: Effect of conditioned and normal air on yarn hairiness (injector diameter: ϕ = 0.8 mm)



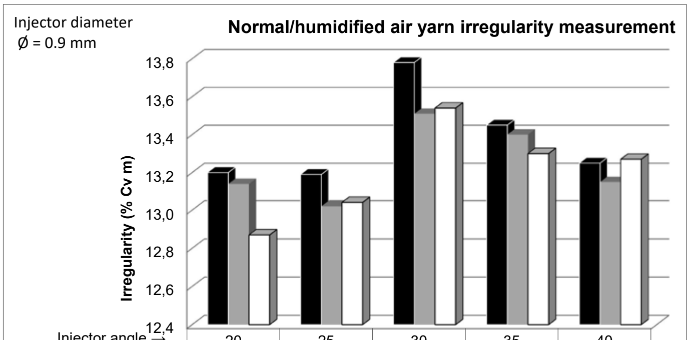
Injector angle →	20	25	30	35	40
■ Normal air	6,29	6,85	6,39	6,14	6,35
■ Humidified air first measurement	6,35	6,84	6,17	6,13	6,58
□ Humidified air second measurement	6,27	6,61	6,24	6,05	6,19

Figure 9: Effect of conditioned and normal air on yarn hairiness (injector diameter: $\phi = 0.9$ mm)



Injector angle →	20	25	30	35	40
■ Normal air	13,03	13,33	12,82	12,98	12,84
■ Humidified air first measurement	12,84	12,99	12,92	12,70	13,58
□ Humidified air second measurement	12,61	13,07	12,91	13,33	12,76

Figure 10: Effect of conditioned and normal air on yarn irregularity (injector diameter: $\phi = 0.8$ mm)



Injector angle →	20	25	30	35	40
■ Normal air	13,20	13,19	13,78	13,45	13,25
■ Humidified air first measurement	13,14	13,02	13,51	13,40	13,15
□ Humidified air second measurement	12,87	13,04	13,54	13,30	13,27

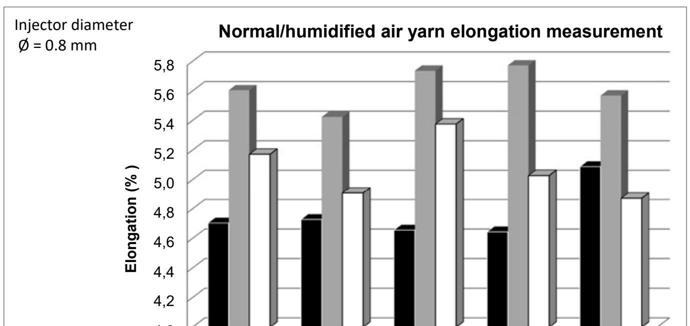
Figure 11: Effect of conditioned and normal air on yarn irregularity (injector diameter: $\phi = 0.9$ mm)

3.3 Yarn elongation results

The effect of conditioned and normal air on yarn elongation (%) is illustrated in Figure 12 for an injector diameter of ϕ = 0.8 mm and in Figure 13 for an injector diameter of ϕ = 0.9 mm. If the second measurement of the humidified nozzle of ϕ = 0.8 mm injector diameter and 40° nozzle is not taken into account, the elongation at break in all samples produced with conditioned air is in the range of 0.5–1% in the first measurement and 0.2–0.8% in the second measurement, where an increase was observed. The reduction in yarn elongation between the first measurement and the second measurement was 0.2% to 0.3%. The humidified air was also observed to improve the yarn elongation property of the yarn, as in the previous results.

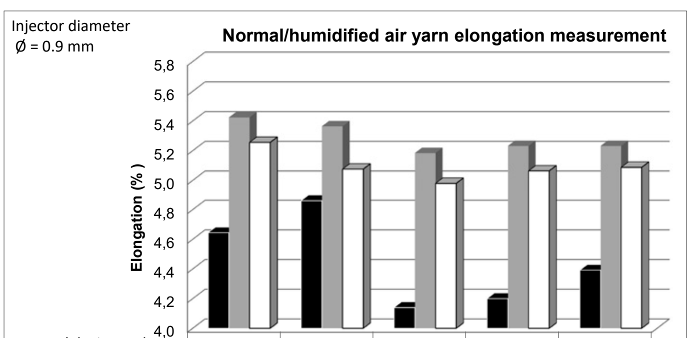
3.4 Yarn tenacity results

The effect of conditioned and normal air on yarn tenacity (cN/tex) is illustrated in Figure 14 for an injector diameter of ϕ = 0.8 mm and in Figure 15 for an injector diameter of ϕ = 0.9 mm. In the nozzle configurations with an injector diameter of ϕ = 0.8 mm, the strength values increased in the first measurement of the humidified air nozzle. In the second measurement of the humidified air sample, however, the strength values were similar to those of normal air. In all other nozzle configurations, except 25° with an injector diameter of ϕ = 0.9 mm, both of the humidified air sample measurements show an average yarn tenacity increase of 0.9–1.8 cN/tex in all samples. Thus, in all other nozzle configurations except 25°, the use of conditioned air results in an increase in yarn tenacity of about 11% to 16%.



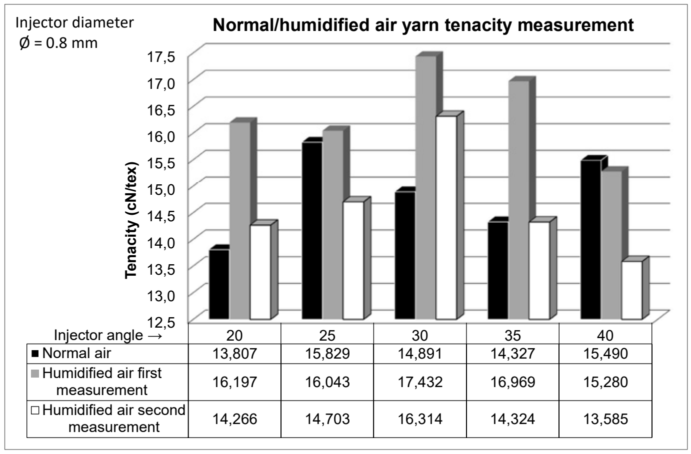
Injector angle →	20	25	30	35	40
■ Normal air	4,704	4,728	4,656	4,644	5,088
■ Humidified air first measurement	5,604	5,424	5,736	5,772	5,568
□ Humidified air second measurement	5,172	4,908	5,376	5,028	4,872

Figure 12: Effect of conditioned and normal air on yarn elongation (injector diameter: ϕ = 0.8 mm)



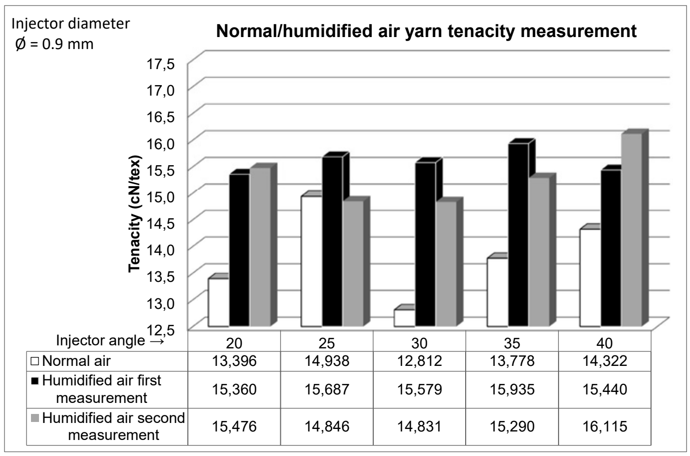
Injector angle →	20	25	30	35	40
■ Normal air	4,644	4,860	4,140	4,200	4,392
■ Humidified air first measurement	5,424	5,364	5,184	5,232	5,232
□ Humidified air second measurement	5,256	5,076	4,980	5,064	5,088

Figure 13: Effect of conditioned and normal air on yarn elongation (injector diameter: ϕ = 0.9 mm)



Injector angle →	20	25	30	35	40
Normal air	13,807	15,829	14,891	14,327	15,490
Humidified air first measurement	16,197	16,043	17,432	16,969	15,280
Humidified air second measurement	14,266	14,703	16,314	14,324	13,585

Figure 14: Effect of conditioned and normal air on yarn tenacity (injector diameter: $\phi = 0.8$ mm)



Injector angle →	20	25	30	35	40
Normal air	13,396	14,938	12,812	13,778	14,322
Humidified air first measurement	15,360	15,687	15,579	15,935	15,440
Humidified air second measurement	15,476	14,846	14,831	15,290	16,115

Figure 15: Effect of conditioned and normal air on yarn tenacity (injector diameter: $\phi = 0.9$ mm)

4 Conclusion

- The use of humidified air on jet-rings had a slight positive effect on all yarn properties (yarn hairiness, yarn irregularity, yarn elongation and yarn tenacity). According to the results, a 1% -3% improvement in yarn quality was achieved.
- In moistened nozzle applications, the second measurement was expected to be regressed or slightly reduced compared to the first measurement. On the contrary, however, an improvement was seen in yarn hairiness and yarn irregularity between the first and second measurements. Yarn elongation and yarn tenacity values decreased slightly as expected.
- In this study, humidification experiments were not performed on different nozzle types. We recommend that researchers work with different types of nozzles in future studies, as there is a higher likelihood of improvement in other types of nozzles.
- During the experiment with the humidified air, there were no yarn breaks due to friction, irregularity or other reasons. Humidified air was also observed to reduce yarn breaks.

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Eco-Friendly Vat Dyeing of Cotton Using Alkaline Iron (II) Salt as Reducing Agent

Okolju prijazno barvanje bombaža z redukcijskimi barvili z uporabo alkalne železove (II) soli kot redukcijskega sredstva

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Abstract

Sodium hydrosulphite is used commercially as the reducing agent for vat dyes in the dyeing of cotton. Large amounts of sodium sulphate, sulphur oxyanion and toxic sulphite are produced during the dyeing due to the dissociation of sodium hydrosulphite leading to severe air and water pollution. This research focuses on the use of alkaline iron (II) salt as the reducing agent for vat dyeing on cotton fabrics through a complete replacement of hydrosulphite. The 3^4 Box-Behnken design was used to achieve optimum parameters and statistically analyse the performance of the new reducing system. The results showed that the alkaline iron (II) salt system was relatively effective in developing a comparable dyebath reduction potential, surface colour strength of cotton and colourfastness, if compared to the hydrosulphite-based reducing system. The dyebath stability in the presence and absence of the dye also showed superior results compared to that of the hydrosulphite system. Hence, it can be said that a complete substitution of sodium hydrosulphite with alkaline iron (II) salt is possible.
Keywords: cotton, vat dyes, hydrosulphite, ferrous sulphate, solubility, colour strength

Izvleček

Natrijev hidrosulfit se tržno uporablja kot redukcijsko sredstvo za redukcijska barvila, ki se uporabljajo za barvanje bombaža. Velika količina natrijevega sulfata, žveplovih oksianionov in toksičnega sulfita, ki nastane pri disociaciji natrijevega hidrosulfita, med barvanjem vodi do močnega onesnaženja zraka in vode. Ta raziskava se osredinja na popolno zamenjavo hidrosulfita z alkalno železovo (II) soljo kot redukcijskega sredstva za barvanje bombažne tkanine z redukcijskimi barvili. Za doseganje optimalnih parametrov in statistično analizo učinkovitosti novega redukcijskega sistema je bila uporabljena metoda 3^4 Box-Behnken. Rezultati so pokazali, da je v primerjavi z redukcijskim sistemom na osnovi hidrosulfita sistem z uporabo alkalne železove (II) soli učinkovitejši pri razvijanju primerljivega redukcijskega potenciala barvne kopeli, jakosti obarvanja bombaža in obstojnosti barv. Stabilnost barvalne kopeli v prisotnosti barvila in njeni odsotnosti je bila v primerjavi s hidrosulfitnim sistemom prav tako boljša. Zato lahko rečemo, da je mogoča popolna zamenjava natrijevega hidrosulfita z alkalno železovo (II) soljo.
Ključne besede: redukcijsko barvilo, hidrosulfit, železov sulfat, topnost, jakost obarvanja

1 Introduction

Dyeing cellulosic fibres with vat dyes provides excellent colourfastness properties in terms of washing, rubbing and light [1–2]. Vat dyes are practically insoluble in water; dyeing is a somewhat complicated procedure, involving reduction and solubilisation steps to attain the water-soluble form of the dye molecule that has high substantivity on cotton. For the reduction of dyes, a reduction potential from around –750 mV to –1000 mV is required for a proper reduction and to retain the reduced form of the dye throughout the dyeing [3–4]. Sodium hydrosulphite is commercially used as the reducing agent in the dyeing of cotton with vat dyes [5]. During the dyeing, large amounts of sodium sulphate, sulphur oxyanion and toxic sulphite are produced, leading to severe air and water pollution. It is also worth emphasising that sodium hydrosulphite cannot be recycled as it mostly gets dissociated in the bath, and the disposal of exhausted dyebaths and rinsing water causes high costs and various problems with the effluent, viz. high salt load, depletion of dissolved oxygen, problems with nasal nuisance etc [6]. To overcome these problems, attempts have been made to develop alternative eco-friendly reducing systems [7]. Such new systems include electrochemical reduction [8], organic reducing agents such as hydroxyacetone [9], iron (II) salt complexes [10], and natural reducing agents such as sheghar [11], orange peel extract [12] and palm wine [13]. In the electrochemical reduction method, a reducing agent must be added to the reduced dyestuff in order to ensure corresponding stability of the reduced liquor. However, the dyestuff requirement to produce a specific shade is higher [14]. Sodium dithionite can be completely replaced with hydroxyacetone and is biocompatible, yet costlier. Iron (II) salts were widely used in ancient times to reduce vat dyes through the technique known as the "copperas method", where ferrous sulphate and calcium hydroxide were used. However, the dyebath resulted in bulky sediments due to the poor solubility of $Fe(OH)_2$. It was found that $Fe(OH)_2$ produced via the reaction of iron (II) salts with sodium hydroxide can be complexed and taken back into the solution to obtain the desired reduction potential value. Iron (II) salts along with the gluconic acid and NaOH at 60 °C, iron (II) salts in combination with the tartaric or citric acid, triethanolamine and NaOH at room temperature and alkaline enzymes are capable of generating the required reduction potential by solubilising $Fe(OH)_2$ generated from iron salts [15–19]. The basic problem associated with use of sodium dithionite as the reducing agent can hence be eliminated by introducing alkaline iron (II) salts. In the case of a natural reducing alternative, it is not possible to use sheghar, orange peel extract and palm wine commercially due to their inherent problems such as extraction via steam distillation plant; furthermore, their huge quantity required for the reduction may rise to additional costs [20].

The reduction of vat dyes using iron (II) salt was earlier attempted by many researchers with no practical success due to the very poor solubility of $Fe(OH)_2$ in water, resulting in an inadequate reduction potential, making the reduction of vat dyes impossible. However, $Fe(OH)_2$ can be solubilised to a higher extent by increasing the concentration of NaOH to generate a very high reduction potential. Only iron (II) salt and NaOH were used in this study, and for a comparison in performance, $Na_2S_2O_4$ was used, which is applied in practice in industries. While iron (II) salt is eco-friendly, NaOH at a higher concentration is not as eco-friendly as iron (II) salt, but is still far better than hydrosulphite. In the conventional $Na_2S_2O_4$ reduction system, NaOH is used as well.

In the present work, the effectiveness of alkaline iron (II) salt was studied regarding the reduction and solubilisation of vat dyes, attempting to substitute sodium hydrosulphite in the vat dyeing technology to formulate a green and eco-friendly process. The extent of the dissolution of the formed $Fe(OH)_2$, the reducing agent which is directly proportional to an increase in the reduction potential of the dyebath at various concentrations of alkali was studied and the relationship of the soluble $Fe(OH)_2$ with a reduction potential of the bath was explored. The solubility pattern of $Fe(OH)_2$ was studied with a rise in temperature to study the rise in the reduction potential of the bath as well. The dyeing performance in terms of the reduction potential at different stages of the dyeing, change in pH, stability of dyebaths in the presence and absence of the dye and the colourfastness of dyed cotton were studied through a statistical analysis.

2 Experimental

2.1 Materials

A commercially scoured, bleached cotton fabric (34.5 ends/cm, 27.4 picks/cm), warp and weft yarn linear density 20.3 tex and 30.2 tex, respectively, was used for the purpose of the research.

Sodium hydroxide (AR, 98%), ferrous sulphate (AR, 98%), hydrogen peroxide (30%), $K_2Cr_2O_7$, diphenylamine, concentrated H_2SO_4 and distilled water were procured from SD Fine chemicals Limited, Mumbai. Ten different vat dyes of commercial grade were obtained for this study from ATIC and Indian Dyestuff Industries, i.e. Green XBN (Vat Green 1, C.I. 59825), Brown BR (Vat Brown 1, C.I. 70800), Yellow 5G (Vat Yellow 2, C.I. 67300), Gold Orange 3G (Vat Orange 15, C.I. 69025), Red 6B/UD (Vat Red 10, C.I. 67000), Grey M (Vat Black 8, C.I. 71000), Brown R (Vat Brown 3, C.I. 69015), Indigo (C.I. Vat blue 1), Blue BC (Vat Blue 6, C.I. 69825) and Olive Green B (Vat Green 3, C.I. 69500).

2.2 Methods

2.2.1 Dyeing of cotton using sodium hydrosulphite

The dyebaths were prepared with a vat dye (1% on the weight of fabric, owf) pasted with T R oil, hot water being added as well. Sodium hydrosulphite (10 g/l) for the reduction and sodium hydroxide (10 g/l) for the solubilisation were added and stirred well until the vatting of the dye took place over a period of about 15 to 20 min at 60 °C. Cotton was dyed for 60 min at 60 °C at 1 : 25 liquor ratio. The bath was then dropped, dyed cotton was cold washed and oxidised with hydrogen peroxide (1–2 g/l) at 50–60 °C for 20–30 min, followed by soaping and a final wash. This was the so-called control sample.

2.2.2 Dyeing of cotton using alkaline iron (II) salt

The dyebaths were prepared with a vat dye (1% owf), iron (II) salt (1–50 g/l) and sodium hydroxide (5–40 g/l) at 80 °C for 90 minutes at 1 : 25 liquor ratio. In this method, NaOH of specific concentration was dissolved in 100 ml of water in a glass beaker to make an alkaline solution followed by the pouring of NaOH to ferrous sulphate ($FeSO_4$) to form insoluble $Fe(OH)_2$ and the bath turned turbid. The quantity of the dye required to get 1 wt% dye was added in this bath to produce 1% shade. Vatting and dyeing were carried out at 80 °C, keeping two parameters constant with varying iron (II) salt and NaOH concentrations. The dyed fabrics were cold washed thoroughly and oxidised with hydrogen peroxide, followed by soaping and washing.

Deciding concentration of $FeSO_4$ and NaOH

$$FeSO_4 + 2NaOH = Fe(OH)_2 + Na_2SO_4 \quad (1)$$

Molecular weight of $FeSO_4 \cdot 7H_2O$ = 278.08 g/mol
Molecular weight of NaOH = 40 g/mol

Equation 1 shows that the reactant 1 mole of ferrous sulphate and 2 moles of NaOH were combined together to form 1 mole of ferrous hydroxide and 1 mole of sodium sulphate. This equation is balanced for the stoichiometric requirement.

278.08 g of $FeSO_4$ require 80 g of NaOH. Similarly, 1 g of $FeSO_4$ requires 0.28 g of NaOH. This means that 0.28 g of NaOH is required for the stoichiometric balance, which has no effect on the alkalinity in the dyebath. The addition of extra NaOH into the dyebath increases alkalinity, which is the major requirement in the alkaline iron (II) salt system. Iron (II) salt (1–50 g/l) was selected based on our previous investigations of iron (II) salt along with enzymes, ligand and sodium hydroxide used in vat dyeing.

2.3 Statistical analysis of dyeing process

The Box-Behnken design was used to study colour strength (*K/S*) as a response for cotton dyed with alkaline iron (II) salt for the optimisation of process parameters. Colour strength (*K/S*) is expressed in terms of the Kubelka-Munk equation as follows:

$$\frac{K}{S} = \frac{\text{Co} - \text{efficient of absorption}}{\text{Co} - \text{efficient of scattering}} = \frac{(1 - R)^2}{2R} \quad (2),$$

where *R* stands for the reflectance of the specimen.

The Box–Behnken design optimises the number of experiments to be performed to ascertain possible interactions between the studied parameters. This included identifying the best suitable combinations of the levels and parameters to achieve a better response equivalent to that obtained in the conventional vat dyeing system. Four factors, i.e. concentrations of iron (II) salt (A), NaOH (B), dyeing temperature (C), time (D), with respective coded values are shown in Table 1.

Using these four factors, each with three levels, the 3^4 Box-Behnken design was run to obtain a set of data, consisting of 27 runs with three replicates at the central point. The Response Surface Methodology (RSM) was applied to the experimental data using statistical software, Design Expert 7. The results were analysed using response surface plots and equations were formed for a response at 95% confidence level. Response surface figures were analysed to understand the effect of each parameter (factor) on colour strength (*K/S*). A regression equation was formed accordingly. A quadratic model with four independent factors was used to analyse

Table 1: Input with coded value

Coded factor	Factors (Independent variables)	Levels		
		(low)	(medium)	(high)
A	Ferrous sulphate	–1	0	+1
B	Sodium hydroxide	–1	0	+1
C	Temperature	–1	0	+1
D	Time	–1	0	+1

the relationship of colour strength (*K/S*). Model accuracy was verified to measure the goodness of fit to the model using the coefficient of determination (R^2). When R^2 approaches unity, the empirical model fits the actual data. P-values lower than 0.05 were considered statistically significant. The lack of fit was analysed to check the model accuracy. The regression equation and response surface figures were used to predict the optimised combination of factors that result in maximum colour strength (*K/S*) at optimised values.

2.4 Evaluation of dyebath and dyed samples

The status of the dyebath reduction was evaluated for the reduction potential as well as for pH at various stages of dyeing, i.e. in a blank reduction bath, after the reduction of the dye and at the end of dyeing, using a digital pH metre and a digital combined oxidation reduction potential (ORP) metre (Century Instruments, India), respectively. Cotton samples dyed in both the hydrosulphite- and alkaline iron (II) salt-based dyebaths were evaluated for colour strength (*K/S*) using Datacolor Check Spectrophotometer (Data-colour International, US). Wash- and lightfastness of dyed cotton were assessed according to respective AATCC standards, i.e. AATCC TM61-2007 (wash) using a wash-fastness tester (RBC Electronics, Mumbai) and AATCC TM16-2004 (light) using an ATIRA lightfastness tester (Paresh Engineering Works, Ahmedabad). The tensile strength of dyed fabrics was assessed according to ASTM D5035 using a universal testing machine (Aimil, Delhi).

2.5 Evaluation of dyebath stability

Reduction baths were prepared with hydrosulphite along with NaOH and alkaline iron (II) salt separately. To study bath stability in the absence of the dye, blank reduction baths were stored for 0 h, 1 h, 2 h, 4 h, 8 h, 12 h and 24 h. The reduction potential of these baths was evaluated after specific time of storage followed by the addition of a Vat Green XBN dye (1% owf) and the dyeing of cotton in these baths. The changes in the reduction potential and pH at each stage were noted. To study the reduction potential of stored reduced dyebaths towards successful dyeing, hydrosulphite- and alkaline iron (II) salt-based reduced dyebaths along with the dye were prepared and stored for up to 24 h prior to the dyeing.

2.6 Estimation of colour strength after HCl treatment

The surface colour strength (*K/S*) of the control sample was evaluated as usual. In iron (II) salt-based baths, iron precipitated on dyed cotton during the dyeing and the hues changed. Precipitated iron was removed from the dyed samples with a treatment with HCl (10% w/v) at 60 °C for 10–15 min. The surface colour strength (*K/S*) was analysed before and after the removal of iron using a computer colour matching spectrophotometer.

2.7 Analysis for solubility of $Fe(OH)_2$

Ferrous hydroxide, $Fe(OH)_2$, is a strong reducing agent; however, it remains mostly insoluble in water [21]. It can be partly solubilised in an alkaline medium though not adequately for the reduction of the dye as the desired solubility can be achieved with an increase in temperature and concentration of alkali. The solubility of ferrous iron present in the reduction bath can be evaluated with titration with a standard solution of potassium dichromate [22].

In titration with a standard solution of $K_2Cr_2O_7$, the volume of the oxidant ($K_2Cr_2O_4$) completely oxidises ferrous iron Fe^{2+} to ferric iron (Fe^{3+}) and $K_2Cr_2O_7$ itself undergoes a reduction due to a high reduction potential, gets converted to Cr^{3+}, which is green in colour even after the equivalence point. To identify the equivalence point, 3–5 drops of diphenylamine (E^0Red = 760 mV were added as an internal indicator [23]. In order to prevent the oxidation of diphenylamine prior to the oxidation of Fe^{2+}, phosphoric acid was added, which reduces the reduction potential of iron couple from 770 mV to 440 mV. Diphenylamine was used as an indicator. At the end, the addition of excess dichromate oxidised diphenylamine to diphenyl benzidine, which developed a deep bluish violet colour.

A standard potassium dichromate solution was prepared by weighing out potassium dichromate (2.45 g) into a 500 ml standard flask and dissolved in a small volume of distilled water to make 0.1 N $K_2Cr_2O_7$ solution. An acid mixture was prepared by mixing up 100 ml of phosphoric acid with 300 ml of conc. H_2SO_4 in a reagent bottle. Diphenylamine solution was prepared by dissolving 1 g of diphenylamine in 100 ml of conc. H_2SO_4. Ferrous iron can be standardised by rinsing the burette with a $K_2Cr_2O_7$ solution and taking the $K_2Cr_2O_7$ solution up to the zero mark of the burette. 10 ml of the ferrous solution was pipetted out into a 250 ml conical flask, 2.5 ml of acid mixture and 1–2 drops of the diphenylamine indicator were added. The resulting solution was titrated with 0.1 N $K_2Cr_2O_7$ until a blue violet colour was obtained.

3 Results and discussion

3.1 Dyeing of cotton with sodium hydrosulphite

Cotton was dyed with ten different vat dyes (1% owf) using hydrosulphite and NaOH (10 g/l each), followed by the oxidation with H_2O_2. The *K/S* of dyed cotton was found to be 22.7 at the maximum wavelength (λ_{max} ~ 630 nm). The range of pH and reduction potential were found to be 12.2–12.8 and –780 to –880 mV, respectively, at various stages of dyeing.

3.2 Dyeing of cotton with alkaline iron (II) salt

To study the reduction potential of the new alkaline iron (II) salt system for successful dyeing of cotton, dyeing factors were initially kept within a certain range in terms of the $FeSO_4$ and NaOH concentration, temperature and time. Colour strength (*K/S*) values were fed into to the Box-Behnken design against different factor-level combinations.

3.2.1 Selection of factors and levels for statistical analysis

To decide on the effective $FeSO_4$ and NaOH concentrations, dyebaths were prepared with a Vat Green XBN dye (1% owf), $FeSO_4$ (1–50) g/l, NaOH (8–50) g/l, temperature of vatting and dyeing remaining constant at 80 °C with the dyeing time of 1 hour. The combined effect of $FeSO_4$ and NaOH on K/S is shown in Table 2; temperature and time were kept constant. The starting NaOH concentration was 8 g/l. The increase in the alkali concentration was responsible for the solubilisation of $Fe(OH)_2$ in the dyebath, which in turn caused an increase in the colour yield (*K/S*). However, beyond the concentrations of sodium hydroxide and $FeSO_4$ at 30 g/l each, there was no further improvement in the colour strength of dyed cotton. Therefore, the $FeSO_4$ concentration at 30 g/l was selected for a further study with a variation in the alkali concentration from 20 to 40 g/l.

To initially assess the effective NaOH concentration, dyebaths were prepared in the same way, i.e. Vat Green XBN dye (1% owf), $FeSO_4$ (30 g/l), NaOH (20–40 g/l), temperature of vatting and dyeing remaining constant at 80 °C with the dyeing time of 1 hour. The effect of the NaOH concentration on *K/S* is shown in Table 3, where it can be seen that the NaOH concentration (level) showed a positive response in a statistical analysis within the range from 26 g/l to 28 g/l.

Table 2: Analysis of effective concentration of $FeSO_4$ and NaOH on K/S

NaOH (g/l)	*K/S* at various concentrations (g/l) of $FeSO_4$						
	1	5	10	20	30	40	50
5	-	-	-	-	-	-	-
10	-	-	-	2	2.4	2.8	2.2
20	-	-	2	5	6	6.5	6.5
30	-	-	2.8	13	16	15.3	13
40	-	-	2	11	13	12.7	13.2
50	-	-	2	11	13	12.8	12

Table 3: Influence of effective NaOH concentration on K/S

NaOH (g/l)	Coloure strength (*K/S*)
20	6
22	8
24	16
26	17
28	16.7
30	16
32	15
34	15.2
36	14.8
38	14.2
40	13

Influence of temperature

The vatting and dyeing temperatures were maintained at 80 °C to see if this has any influence on *K/S*. It was observed that the *K/S* increases with an increase in temperature from 30–90 °C (Figure 1). Dyeing is usually an exothermic process, i.e. heat releasing. The interaction between the dye and fibre is stronger than that of between the dye and water molecules in a solution. As the dyeing temperature increases, aggregates gradually break up through the absorption of heat; hence, more individual molecules are available for a penetration into the fibres. The exhaustion thus increased with an increase in temperature very slowly from 30–40 °C, beyond which the rate of the increase was uniformly high from 40–90 °C. The increase in colour strength with temperature was mainly a consequence of the breaking down of dye aggregates and the increase in the solubility of $Fe(OH)_2$.

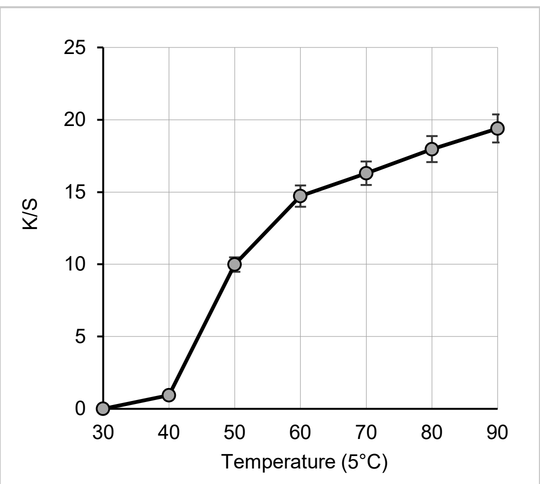

Figure 1: Effect of temperature on surface colour strength (K/S)

Influence of time

The time of dyeing was 30 min, 60 min, 90 min, 120 min, 150 min and 180 min, with other parameters remaining unchanged. The effect of the dyeing time on *K/S* is shown in Figure 2. It was observed that *K/S* increased with an increase in the dyeing time up to 60 min beyond which there was no improvement in *K/S* till 120 min, succeeded by progressive falling probably due to an increase in the stripping of dyes. Consequently, for a further statistical study, the selected range of the dyeing time was 60 min, 90 min and 120 min.

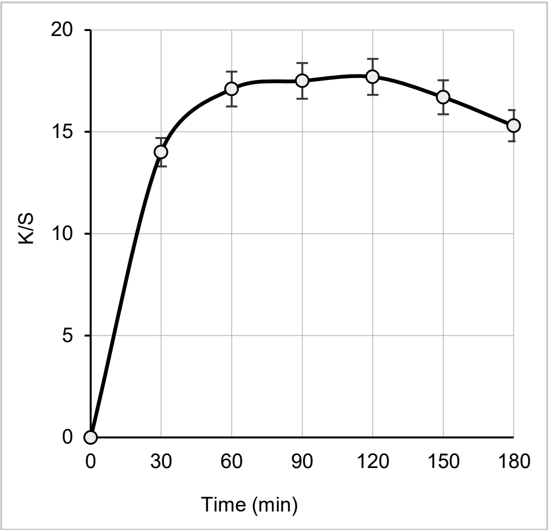

Figure 2: Influence of time on colour strength

3.2.2 Statistical analysis

The Box-Behnken design was run with the factors and levels of different parameters (cf. Table 4), thus generating 27 runs (cf. Table 5). A cotton fabric was dyed in these baths in the same manner as used in the $Na_2S_2O_4$ system and the *K/S* of the dyed specimen was evaluated. It was observed that the range of K/S was 12.6–21.8 and the highest K/S was observed if the bath was prepared according to the 27th run, i.e. $FeSO_4$ (30 g/l), NaOH (27 g/l), temperature (90 °C) and time (120 min) as detailed in Table 5.

The dyed specimen obtained through the run no. 27 showed the maximum *K/S* value at higher temperature and longer time, whereas the run no. 10 showed a slightly lower *K/S* value at shorter time, which would be preferred from the economical point of view. Obviously, the parameters of the run no. 10 were selected for a further study.

Table 4: Factors and actual levels for dyeing with alkaline iron (II) salt

Coded factor	Factors (Independent variables)	Level		
		Low (–1)	Medium (0)	High (+1)
A	$FeSO_4$ (g/l)	25	30	35
B	NaOH (g/l)	26	27	28
C	Temperature (°C)	60	75	90
D	Time (min)	60	90	120

Table 5: Box-Behnken experimental design layout and observed response

Run	$FeSO_4$ (g/l)	NaOH (g/l)	Temperature (°C)	Time (min)	*K/S*	Standard deviation
1	30	28	75	120	14.2	0.2
2	35	28	75	90	15.2	0.264
3	30	27	75	90	17.2	0.264
4	30	27	60	120	13.9	0.251
5	35	27	75	120	18.6	0.152
6	30	26	60	90	13.8	0.173
7	35	27	60	90	12.9	0.208
8	25	27	60	90	14.5	0.1
9	25	27	75	120	16.5	0.2
10	30	27	90	60	**21.2**	0.115
11	30	27	75	90	18.6	0.288
12	30	27	75	90	18.8	0.1
13	30	28	60	90	14.8	0.3
14	30	28	90	90	19.6	0.1
15	35	26	75	90	20.2	0.1
16	25	27	75	60	14.6	0.173
17	30	26	90	90	21.4	0.351
18	35	27	75	60	17.4	0.152
19	25	28	75	90	16.8	0.4
20	25	26	75	90	15.4	0.3
21	30	28	75	60	14.9	0.1
22	30	27	60	60	12.6	0.2
23	35	27	90	90	21.6	0.3
24	25	27	90	90	20.1	0.057
25	30	26	75	60	13.6	0.1
26	30	26	75	120	19.6	0.3
27	30	27	90	120	**21.8**	0.2

3.3 Influence of dyeing parameters on K/S

ANOVA is a statistical technique that subdivides the total variation in a set of data into component parts associated with specific sources of variation to test hypotheses on the parameters of the model. The Box-Behnken design was used to analyse the effect of process parameters, i.e. concentrations of $FeSO_4$, NaOH, and temperature and time, on *K/S*. All main effects, two factors interaction and the cubic effect with $R^2 \sim 0.94$ obtained using ANOVA are presented in Table 6. R^2 represents the proportion of variation in the response data that can be explained by the fitted model. Therefore, the higher the R^2, the better the model. The model was significant at 95% confidence level, when the value of probability > F' was less than 0.05. The ANOVA table also shows a term of residual error, which measures the amount of variation in the response data left unexplained by the model. The lack of fit term in the residual indicates the variation due to model inadequacy. All p-values for the lack of fit (larger than 0.05 for all responses) indicate that the model adequately fits the data. In other words, the form of the model chosen to explain the relationship between the factors and the response is correct and all the prerequisites associated with the use of ANOVA are met as well. The concentrations of $FeSO_4$, NaOH, temperature and time were significant model factors, the model equation in coded form being shown in equation 3.

$$K/S = -1248 + 9.32 \times A + 73.89 \times B + 1.19 \times C + 1.82 \times D - 0.32 \times A \times B + 0.01 \times A \times C - 1.16 \times A \times D - 0.04 \times B \times C - 0.05 \times B \times D - 3.88E{-}004 \times C \times D - 0.02 \times A^2 - 1.04 \times B^2 - 1.85E{-}004 \times C^2 - 1.24E{-}003 \times D^2 \quad (3),$$

where different values of A, B, C, D are presented in Table 4. A is the dimensionless number taken from Table 4 neglecting the unit, i.e. considering the absolute minimum values, A = 25, B = 26, C = 60 and D = 60. The same is true for the intermediate and maximum dimensionless values of A, B, C and D as shown in Table 4.

This equation can predict the theoretical *K/S* of dyed specimens for given dyeing parameters. Subtracting non-significant factors from equation 3 having p-value beyond 0.5, i.e. AD, CD and C^2, the final equation stands to:

$$K/S = -1248 + 9.32 \times A + 73.89 \times B + 1.19 \times C + 1.82 \times D - 0.32 \times A \times B + 0.01 \times A \times C - 0.04 \times B \times C - 0.05 \times B \times D - 0.02 \times A^2 - 1.04 \times B^2 - 1.24E{-}003 \times D^2 \quad (4).$$

Table 6: ANOVA table for K/S

Source	Sum of squares	df	Mean square	F value	p-value Prob > F	Significance
Model	213.42	14	15.24	14.69	**< 0.0001**	Yes
A – Iron(II)salt	5.33	1	5.33	5.14	**0.0427**	
B – NaOH	6.02	1	6.02	5.80	**0.0330**	
C – Temp.	155.52	1	155.52	149.83	**< 0.0001**	
D – Time	8.84	1	8.84	8.52	**0.0129**	
AB	10.24	1	10.24	9.87	**0.0085**	
AC	2.40	1	2.40	2.31	0.1541	
AD	0.12	1	0.12	0.12	0.7371	
BC	1.96	1	1.96	1.89	0.1945	
BD	11.22	1	11.22	10.81	0.0065	
CD	0.12	1	0.12	0.12	0.7371	
A^2	1.38	1	1.38	1.33	0.2717	
B^2	5.83	1	5.83	5.62	**0.0354**	
C^2	9.259E-003	1	9.259E-003	8.920E-003	0.9263	
D^2	6.70	1	6.70	6.45	**0.0259**	
Residual	12.46	12	1.04			
Lack of fit	10.94	10	1.09	1.44	0.4783	No
Pure error	1.52	2	0.76			
Cor total	225.88	26				

3.3.1 Influence of $FeSO_4$ and NaOH concentrations on K/S

The combined effect of $FeSO_4$ and NaOH concentrations on *K/S* at moderate time and temperature is shown in Figure 3a. A lower concentration of NaOH (26 g/l) and high concentration of $FeSO_4$ (35 g/l) resulted in maximum *K/S*, i.e. 21.8. At a lower concentration of NaOH, decreasing the concentration of $FeSO_4$ resulted in a decrease in the colour strength (*K/S*) of cotton. At a higher concentration of NaOH, there was an increase in the *K/S* value up to a certain limit after which it decreased with a further increase in the $FeSO_4$ concentration. An increased concentration of $FeSO_4$ might decrease the concentration of NaOH in the dyebath due to the formation of insoluble $Fe(OH)_2$ affecting the solubility of the reduced vat. A lower concentration of NaOH had a positive effect on *K/S*. Additional NaOH might resist the diffusion of particles through fibre pores.

3.3.2 Influence of $FeSO_4$ and temperature on K/S

The combined effect of $FeSO_4$ and temperature on *K/S* at moderate NaOH and time is shown in Figure 3b. A higher $FeSO_4$ concentration and temperature resulted in maximum *K/S*, i.e. 21.8. At lower temperature, decreasing the $FeSO_4$ concentration, the *K/S* value increased, whereas at high temperature, decreasing the $FeSO_4$ concentration resulted in decreased *K/S*. At lower temperature, the activation energy of dyeing was too low to solubilise ferrous hydroxide, resulting in decreased *K/S*.

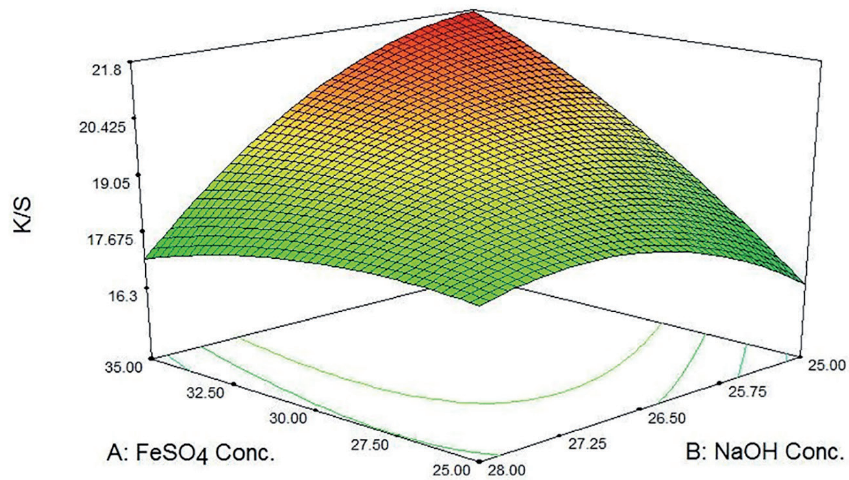

Figure 3a: Effect of interaction of iron (II) salt and NaOH on K/S

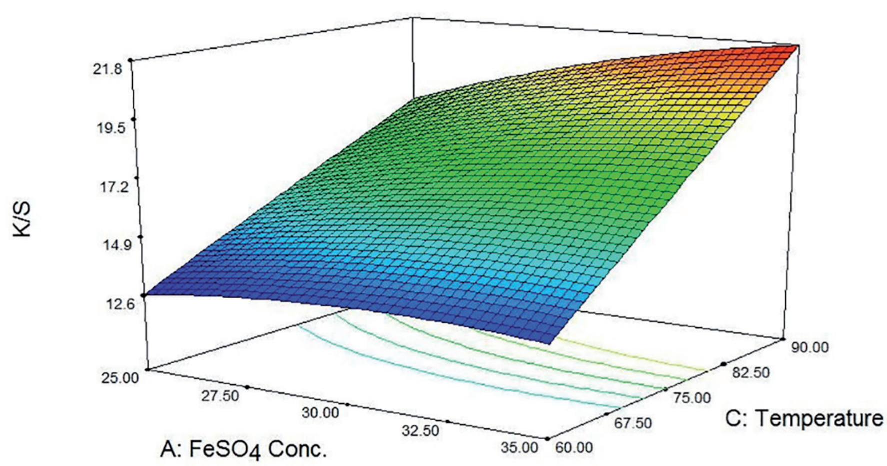

Figure 3b: Effect of interaction of iron (II) salt and NaOH on K/S

3.3.3 Influence of $FeSO_4$ and time on K/S

The combined effect of $FeSO_4$ and time on *K/S* at moderate NaOH and temperature is shown in Figure 3c. A higher concentration of $FeSO_4$ and moderate time resulted in maximum *K/S*, i.e. 21.8. At a higher as well as at a lower concentration of $FeSO_4$, the *K/S* value showed an increasing trend for moderate time and went further on decreasing. At shorter and longer times, the *K/S* value showed an increasing trend with an increase in the concentration of $FeSO_4$. At a higher concentration of $FeSO_4$, the vat dye completely diffused into fibre pores at moderate time; additional time may further reduce this effect. Longer dyeing time may possibly reduce the activation energy required for the absorption and fixation of the dye on fibre surfaces.

3.3.4 Influence of NaOH and time on K/S

The combined effect of NaOH and time on *K/S* at moderate concentration of $FeSO_4$ and temperature is shown in Figure 3d. A low concentration of NaOH and longer time resulted in maximum *K/S*, i.e. 21.8. At shorter time with increased NaOH concentration, there was an increase in the *K/S* value up to a certain limit succeeded by a decrease regardless of a further increase in the NaOH concentration. At longer time, there was a decrease in the *K/S* value with an increase in the NaOH concentration. At a lower concentration of NaOH, there was an increase in *K/S* with time and at a higher concentration of NaOH, there was a decrease in *K/S* with time. A longer time of the dyebath by decreasing the alkali concentration may achieve the desired reduction potential that increases colour strength.

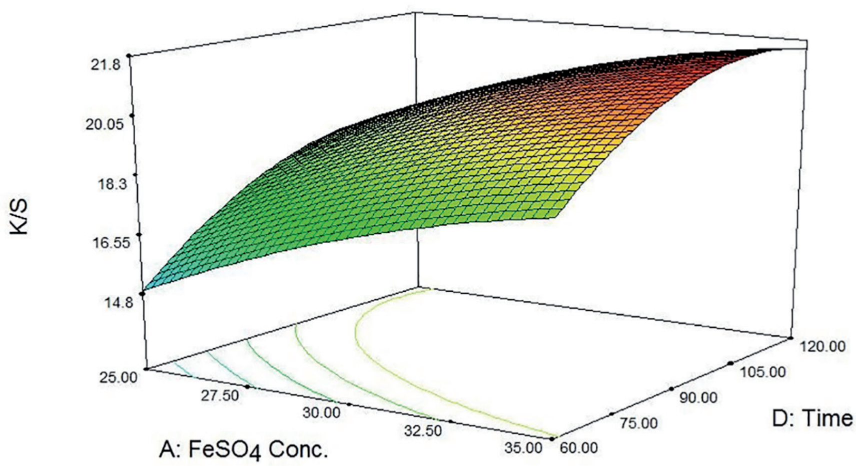

Figure 3c: Effect of interaction of iron (II) salt and time on K/S

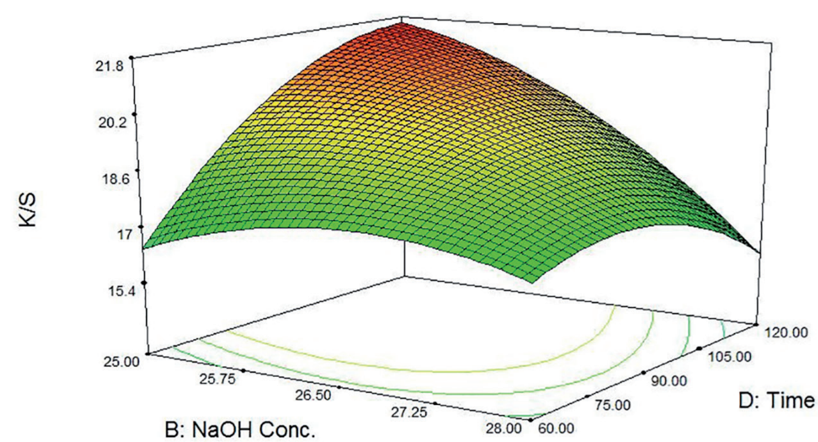

Figure 3d: Effect of interaction of NaOH and time on K/S

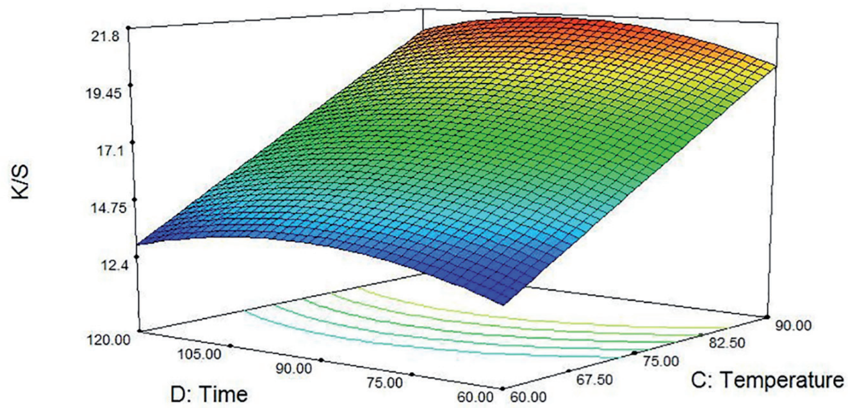

Figure 3e: Effect of interaction of time and temperature on K/S

3.3.5 Influence of temperature and time on K/S

The combined effect of temperature and time on *K/S* at a moderate concentration of $FeSO_4$ and NaOH is shown in Figure 3e. Higher temperature and moderate time resulted in maximum *K/S*, i.e. 21.8. At higher as well as at lower temperature, there was an increase in the *K/S* value up to a certain point of time after which it decreased despite a further increase in time. A longer time period at higher temperature may take additional water due to the exhaustion of the dyebath, which changes ferrous iron to ferric iron, resulting in reduced reduction potential. For a shorter as well as a longer amount of time, there was an increase in the *K/S* value with an increase in temperature.

3.4 Influence of NaOH on reduction potential and pH

In the alkaline iron (II) salt system, 0.84 g of NaOH was taken for the stoichiometry requirement. At that time, pH was neutral as well. A further addition of extra NaOH was responsible for an increase in the solubility of ferrous hydroxide, which increased the reduction potential. $Fe(OH)_2$ solubility increased with an increase in the alkalinity at a certain limit. It was calculated that 3 g Iron (II) salt required 0.84 g of NaOH for the stoichiometric requirement. Additional NaOH was used to maintain the alkaline medium. In this study, 1.8 g NaOH was used for solubilisation. The amount of sodium hydroxide varied in the reaction medium from 0.15 g to 5.65 g. The effect of this variation on the evolution of the redox potential of the medium and pH was investigated. The experimental results are shown in Figure 4.

It can be observed that at sodium hydroxide amounting to 0.15 g to 0.75 g, there was a slow increase in the redox potential and pH, respectively. The increase in the amount of sodium hydroxide led to a rapid jump of the redox potential and pH. At a further addition of 2.25 g of NaOH, the reduction potential and pH remained quasi stable. Therefore, it appears that the alkali addition increased the rate of the vat reduction with iron (II) salt.

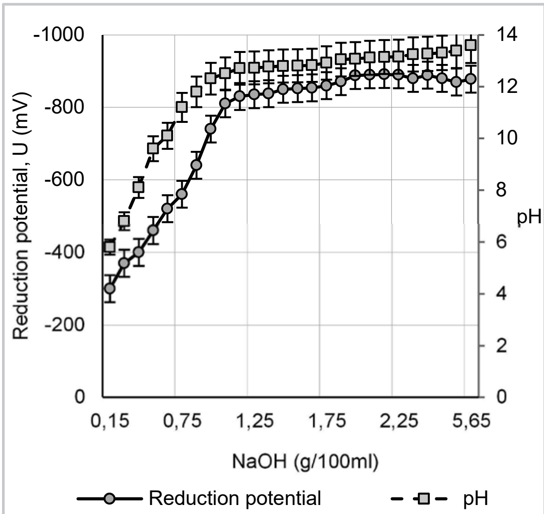

Figure 4: Effect of alkali concentration on pH and reduction potential of dyebath

3.5 Stability of reduction baths

3.5.1 In absence of dye

The stability of reduction baths was studied in the absence of the dye for up to 24 hours with hydrosulphite and the alkaline iron (II) salt system. Reduction baths were prepared and covered; the reduction potential and pH of the baths were checked after being stored for a specific period of time. Vat

Table 7: Stability of reduction baths in absence of dye

Time (h)	Reduction potential, U (mV) and pH at various stages											
	Before reduction of dye				After reduction of dye				After completion of dyeing			
	Hydro		Iron II		Hydro		Iron II		Hydro		Iron II	
	pH	U (mV)	pH	U (mV)	pH	U (mV)	pH	U (mV)	pH	U (mV)	pH	U (mV)
0	12.84	–872	13.95	–708	12.70	–760	13.83	–705	12.40	–748	13.71	–665
1	12.84	–865	13.94	–718	12.58	–755	13.85	–705	12.28	–750	13.69	–633
2	12.83	–870	13.96	–711	12.39	–767	13.84	–648	12.19	–720	13.70	–639
4	12.82	–782	13.93	–705	12.60	–740	13.83	–646	12.20	–730	13.65	–634
8	12.80	–706	13.94	–713	12.40	–685	13.84	–660	12.34	–640	13.67	–620
12	12.82	–680	13.94	–704	12.54	–660	13.82	–655	12.32	–610	13.68	–620
24	12.86	–655	13.92	–696	12.40	–620	13.81	–641	12.27	–580	13.65	–616

Green XBN (1%) was then added to the baths. The dyeing was carried out, with the results presented in Table 7. There was a progressive drop in U (mV) and pH in both reduction baths with the passage of time. *K/S* gradually decreased with an increase in storage time in both reduction systems. Hydrosulphite-based reduction baths retained the required reduction potential for up to four hours compared to the alkaline iron (II) salt-based reduction baths in 24 hours.

3.5.2 In presence of dye

Reduction baths with hydrosulphite and alkaline iron (II) salt were prepared, followed by the addition of Vat Green XBN (1%). The baths were covered and stored for up to 24 hours. The results were noted in terms of pH and reduction potential, and are presented in Table 8.

The rate of the fall in the reduction potential was higher in the hydrosulphite system. In contrast, alkaline iron (II) salt-based systems showed a very slow fall in the reduction potential. However, pH remained in both systems at a higher level. Reduced dyebaths showed the maximum colour strength for dyeing at 0–2 hours (cf. Table 9). The hydrosulphite bath showed stability for up to four hours compared to that in the alkaline iron (II) system for 24 hours with a progressive fall in colour strength with time.

3.6 Fastness performance

The colourfastness of cotton dyed in the hydrosulphite and alkaline iron (II) salt systems was evaluated. The results are shown in Table 10. The lightfastness of dyed cotton remained very good to excellent and almost the same in both systems for the ten dyes

Table 8: Stability of reduction baths in presence of dye

Time (h)	Reduction potential, U (mV) and pH at various stages											
	Before dye reduction				After dye reduction				After completion of dyeing			
	Hydro		Iron II		Hydro		Iron II		Hydro		Iron II	
	pH	U (mV)	pH	U (mV)	pH	U (mV)	pH	U (mV)	pH	U (mV)	pH	U (mV)
0	12.74	–850	13.64	–780	12.60	–865	13.53	–790	12.30	–810	13.34	–770
1	12.74	–810	13.58	–765	12.38	–825	13.46	–775	12.18	–770	13.26	–760
2	12.73	–770	13.47	–760	12.29	–775	13.40	–760	12.09	–760	13.35	–720
4	12.72	–740	13.55	–740	12.5	–735	13.45	–745	12.10	–640	13.29	–730
8	12.70	–690	13.51	–730	12.30	–670	13.36	–740	12.19	–520	13.32	–697
12	12.72	–500	13.55	–725	12.34	–450	13.48	–735	12.12	–400	13.42	–695
24	12.66	–450	13.50	–710	12.2	–400	13.35	–720	12.05	–350	13.24	–685

under study, except for the Yellow 5G dye. Wash fastness, and dry and wet crock-fastness results showed identical, excellent fastness properties for both the hydrosulphite and alkaline iron (II) salt systems, except for the Brown R dye. Overall, the colourfastness of cotton dyed using the alkaline iron (II) salt systems showed comparable performance with the cotton dyed with the hydrosulphite system.

Table 9: Effect of storage time on K/S in presence and absence of dye

Time (h)	Colour strength (*K/S*)			
	Sodium hydrosulphite		Alkaline iron (II) salt	
	Presence of dye	Absence of dye	Presence of dye	Absence of dye
0	22.7	22.6	21.4	21.2
1	20.2	22.7	21.4	18.9
2	18.9	20.8	22.12	18.4
4	15.9	16.4	20.68	16.43
8	0	0	19.46	16.2
12	0	0	17.8	15.9
24	0	0	14.5	13.4

Table 10: Colourfastness of cotton dyed in hydrosulphite and alkaline iron (II) salt systems

Vat dye	Reducing system	Wash fastness			Crock fastness		Light fastness
		Colour change	Stain on cotton	Stain on wool	Dry	Wet	
Brown BR	Hydrosulphite	5	5	5	5	4–5	7
	Alk. iron (II)	5	5	5	5	4–5	7
Yellow 5G	Hydrosulphite	5	5	5	5	5	5
	Alk. iron (II)	5	5	5	5	5	5
Gold Orange	Hydrosulphite	5	5	5	5	5	7
	Alk. iron (II)	5	5	5	5	5	7
Red 6B	Hydrosulphite	5	5	5	5	5	7
	Alk. iron (II)	5	5	5	5	4–5	7
Grey M	Hydrosulphite	5	5	5	5	5	7
	Alk. iron (II)	5	5	5	5	4–5	7
Brown R	Hydrosulphite	5	5	5	5	5	8
	Alk. iron (II)	5	5	5	5	4–5	7
Indigo	Hydrosulphite	5	5	5	5	4–5	7
	Alk. iron (II)	5	5	5	5	4–5	7
Blue BC	Hydrosulphite	5	5	5	5	4–5	8
	Alk. iron (II)	5	5	5	5	4–5	8
Olive Green B	Hydrosulphite	5	5	5	5	5	7
	Alk. iron (II)	5	5	5	5	5	7
Green XBN	Hydrosulphite	5	5	5	5	4–5	8
	Alk. iron (II)	5	5	5	5	4–5	8

3.7 Comparison of colour strength of treated samples

A comparison was made of the colour strength of the samples dyed with different vat dyes with hydrosulphite, alkaline iron (II) salt system and HCl-treated after the soaping in the alkaline iron (II) salt system. Figure 5 shows that all these dyes showed comparable colour strength in the hydrosulphite and alkaline iron (II) salt system though the HCl-treated samples showed slightly lower colour strength.

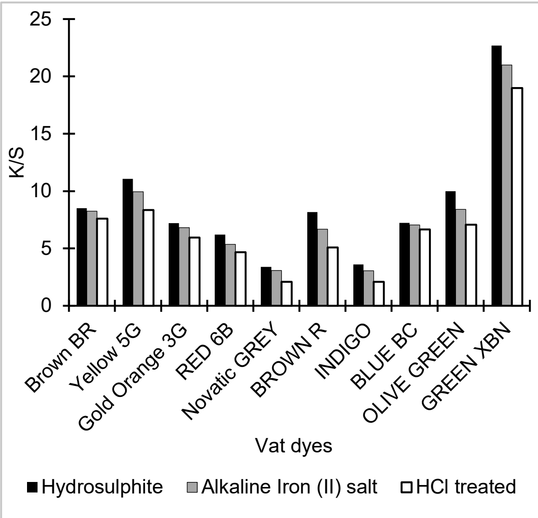

Figure 5: Comparison of K/S obtained as control, reference and HCl-treated

In addition to the result without an HCl treatment, there was a slight difference in colour strength between the control and reference sample. The alkaline iron (II) salt system might be used as an alternative reducing agent; however, it gives a non-uniform deeper shade, indicating the deposition of the insoluble dye and insoluble iron on samples. For a brighter and uniform shade, an HCl treatment is required.

3.8 Solubility of $Fe(OH)_2$

3.8.1 Effect of pH

The effect of alkali on the solubility of $Fe(OH)_2$ is determined by titrating the standard dyebath solution with $K_2Cr_2O_7$. $Fe(OH)_2$ is insoluble at pH 7; a gradual addition of NaOH increases pH, resulting in the solubility of $Fe(OH)_2$. The optimised concentration of alkaline iron (II) salt was taken and pH varied from 7 to 14 by adding NaOH and increasing the solubility of $Fe(OH)_2$ by up to 1.95 g/l as shown in Table 11.

Table 11: Effect of pH on solubility

pH	Solubility of $Fe(OH)_2$ (g/l)
7	0.0143
8	0.27
9	0.55
10	0.83
11	1.11
12	1.39
13	1.67
14	1.95

3.8.2 Effect of temperature in alkaline medium

The solubility of $Fe(OH)_2$ only by adding NaOH was not adequate for the reduction of the vat dye. At low temperature, the heat or chemical energy is not sufficient for the diffusion or penetration of the dye into fibre pores. The increase in temperature is essential to synergise the reduction potential. The effect of temperature on solubility in an alkaline medium was hence studied.

In Figure 6, it is shown that with an increase in temperature in an alkaline medium, the solubility of $Fe(OH)_2$ increased, showing a better result in colour strength. An incomplete vat dye reduction was found at lower temperature and a complete dye reduction at higher temperature. At 30 °C, the solubility of $Fe(OH)_2$ was 1.95 g/l compared to that at 90 °C, which was 3.07 g/l. The amount of total $Fe(OH)_2$ at iron (II) salt 30 g/l and NaOH 27 g/l was 23.4 g/l, where the amount of soluble $Fe(OH)_2$ in the dyebath was 3.07 g/l, the rest remaining insoluble.

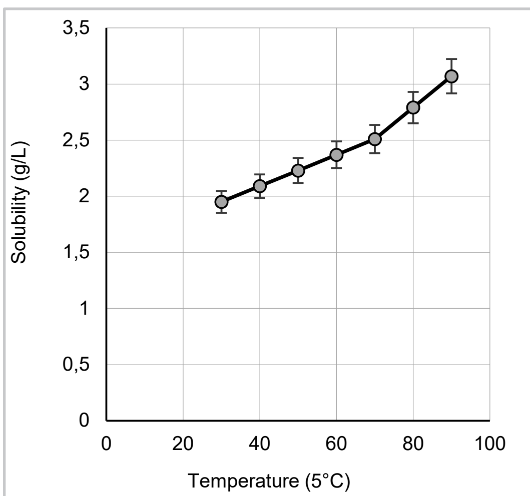

Figure 6: Effect of temperature in alkaline medium on solubility of $Fe(OH)_2$

4 Conclusion

In this study, alkaline iron (II) salt was used to replace sodium hydrosulphite in the vat dyeing of cotton. Iron (II) salt forms $Fe(OH)_2$ in a reaction with NaOH; the former may act as a strong reducing agent. However, due to a very poor solubility of $Fe(OH)_2$ in lower alkaline pH, its performance as a reducing agent cannot be realised. An increase in the concentration of NaOH increases the solubility of $Fe(OH)_2$, thus approaching to a concentration of both chemicals, i.e. $FeSO_4$ and NaOH, to attain the reduction potential as high as –780 mV, showing the capability to reduce all vat dyes. An increase in temperature enhances the reduction potential of the bath as well. The surface colour strength of cotton remained almost comparable along with colourfastness. The reduction bath stability in the absence and presence of the dye shows maximum performance of hydrosulphite-based baths up to maximum storage time of 4 hours and no dyeing beyond that compared to the stability of alkaline iron (II) salt-based reduction baths till 24 hours with a slight progressive fall in colour strength after 8 hours. Moreover, after the removal of precipitated iron from dyed cotton, a small drop in surface colour strength was observed. It can be summarised that the alkaline iron (II) salt-based reducing system can be a good replacement for hydrosulphite-based vat dyeing processes.

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