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Contact:
Ege University, Textile and Apparel Research-Application Center, 35100, Bornova, İzmir, Turkey
Tel: +90 232 311 38 89-88 / Fax: +90 232 311 10 97
(Ege Üni. Tekstil ve Konfeksiyon Araştırma-Uygulama Merkezi 35100 Bornova – İzmir)

www.dergipark.gov.tr/tekstilvekonfeksiyon
E-mail: tekstilkonfeksiyon@mail.ege.edu.tr

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(REFEREED RESEARCH)

THE EVALUATION OF BALKAN COUNTRIES CLOTHING TRADE WITH EU-28 BASED ON THE ANALYSIS OF COMPARATIVE ADVANTAGES INDICES

Sunhilde CUC, Simona TRIPA

University of Oradea, Department of Textile Leather and Industrial Management, Str. B. Şt. Delavrancea, no. 4, 410058, Oradea, Bihor, România

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ABSTRACT

This paper presents an analyses of the trade competitiveness of the clothing sector of Balkans countries' vis-à-vis the EU-28 using 3 indicators: the Balassa index (RCA), the Vollrath's revealed competitiveness (VRC), and Lafay Index (LFI), during 2000–2015. All analyzed countries experienced a decrease in their comparative advantage, though it still remained on a high level in the case of Turkey, Macedonia, and Albania in on acceptable level in the case of Romania. We observed weakening comparative advantage stability, underpinned by convergence of revealed comparative advantage pattern.

Keywords: Competitiveness, comparative advantage, clothing industry

Corresponding Author: Simona Tripa, tripasimona@yahoo.com

INTRODUCTION

Traditionally, textile and clothing industry has been playing a significant role in the economic development and foreign trade of the Balkans. The geographical distribution of production in the garment industry has changed considerably in the past decades resulting in significant economic losses in Europe and North America and important gains especially in Asia. The question is how the Balkans have been influenced by the globalization of clothing production, given that the textile and clothing industries is considered to be traditional for this region. The Balkans is economically the least developed region of Europe, and is considerably differentiated within itself by level of economic development. All Balkan countries had to face challenges such as restructuring of economic system, changing trade markets and patterns, reduction of competitive ability, contracted export base, and lower economies of scale.In presented context, we considered opportune to study and compare the current place of Balkan countries on the clothing European market.

The main aim of this paper is to measure and compare the trade competitiveness of the clothing industry of Balkans countries' vis-à-vis the EU-28 using 3 indicators: the Balassa index (RCA), the Vollrath's revealed competitiveness (VRC), and Lafay Index (LFI), during 2000–2015. The results of this research will help business and policymakers by highlighting the trends in structural change in the clothing trade in each of the observed countries.

Methods of the scientific research that have been employed in the paper are literature survey, mathematic calculations, and comparative analysis of statistic indexes. To provide background for the analysis, the difference between the concepts of comparative advantage and competitiveness is presented briefly in the first section. The second section is about methodology, methods and data used; indices of comparative advantage are developed to examine trade competitiveness of the clothing industry of Balkans countries' vis-à-vis the EU-28. The final part draws some conclusions based on the findings and recommendations.

1. COMPARATIVE ADVANTAGE AND COMPETITIVENESS

The theory of comparative advantage is old but still relevant to compare a country's factor endowment structures; their trade patterns and give some predictive indicators. The concept of comparative advantages has the foundation in trade theory and is usually used in modern economic literature to describe the basic economic benefits that countries get from trading with one another. Comparative advantage occurs if a country is relatively better in producing a specific product than other countries. Given limited resources, a country's choice to specialize in the production of particulars goods is basically influenced by its comparative advantage. There are numerous models that are used to analyze the source of comparative advantage, in this paper we focus on the two most representative: the classical model, developed by Davis Ricardo and the Heckscher–Ohlin model (H–O model), developed by the Swedish economists Eli Heckscher and Bertil Ohlin (see table 1) (1, 2, 3)

Table 1. Ricardian Model vs Heckscher-Ohlin Model

Ricardian Model (Classical Theory)	Heckscher-Ohlin Model (Neoclassical Theory)
2-2-1 Model (2 countries-2 commodities-1 factor)	2-2-2 Model (2 countries-2 commodities-2 factors)
There is only one factor of production: labor.	There are two factors of production: labor and capital.
Is expressed in terms of labor theory of value	Is expressed in terms of money/price theory
The difference in the cost ratios between countries is due to the difference in the skill and efficiency of labor	The relative differences in factor endowments are the causes for the differences in the prices of produces.
The principle of comparative costs is applicable only to international trade.	The principle of comparative costs is applicable to all trade; whether internal or international.
The welfare aspect of the trade is more significant.	The cause of international trade is more important than its welfare aspect.
The model applies in the short-term because the technology can change internationally over time.	The model presume that in the long-term, countries have the same technology.
Countries should specialize in producing goods that they can do best.	Countries should produce and export goods using the resources that they have in abundance.

Source: own representation of the main theories (1, 2, 3)

Therefore, a country has a comparative advantage in the producing those products which uses the relatively abundant resource in that country more intensively and it should specialize in producing goods that it can do best. Over time, the H-O model has been developed by many economists by dropping some of its simplifying hypothesis and acknowledging the differences, however, without changing the fundamental role of variable factor proportions in international trade. There was add to the model some practical considerations (such as tariffs, production technology, and factor price differences, consumption, productivity) to increase the model's predictive power, or as a mathematical way of discussing macroeconomic policy options. A country's comparative advantage in a product can change over time due to changes in any of the determinants of comparative advantage including resource endowments, technology, demand patterns, specialization, business practices, and government policies.(4)

The concept of competitiveness has many interpretations; some scholars use the term synonymously or in a similar way as the comparative advantage while others view it as an economy-wide characteristic. However, competitiveness always involves comparisons between companies or industries in different countries.

While theories defining the comparative advantage have been developed, the question that arises in this context is how to apply the theory in determining the comparative advantage of countries. The concepts of competitive advantage as the basis for the measurement of competitiveness were introduced by Liesner (1958) (5) but refined and popularized by Bela Balassa (1965, 1977) (6) in terms of the revealed comparative advantage (RCA), known as Balassa's index. On the basis of Balassa's index, a country is specialized in exports of a certain product if its market share in that product is higher than the average or, equivalently, if the weight of the product of the country's exports is higher than its weight of the exports of the reference area. Based on the basic concept of revealed comparative advantage, many different RCA indices have been suggested. Vollrath (1991) offered alternative measures of revealing a comparative advantage which include the effects of both the imports and exports of a country. (7). The Lafay index (LFI) shows the alternative measures of specialization, with taking into account both exports and imports flow. It allows analyzing the position of a specific product within the foreign trade structure of every analyzed country. (8)

Table 2. Indicators of comparative advantage

Index	Formula	Meaning
Balassa's Revealed Comparative Advantage Index/ RCA	$RCAij = (Xij / Xit) / (Xnj / Xnt)$ Where: X-exports, i -the country, j-commodity/industry, n-world or a set of countries t- all product groups.	RCAij>1 indicates country i has a comparative advantage in production of j; the greater the index, the stronger the advantage. RCAij<1 indicates that country i has a comparative disadvantage in production of j; the smaller the index, the greater the disadvantage.
Vollrath's Revealed Competitiveness Index (VRC)	$RCij = ln\ (RXAij) - ln\ (RMAij)$ Relative export advantage: *RXA* $RXAij = (Xij / Xit) / (Xnj / Xnt)$ Relative import advantage :*RMA* $RMAij = (Mij / Mit) / (Mnj / Mnt)$	A positive value of the indices reveals a comparative advantage, while a negative value reveals a comparative disadvantage.
Lafay Index /LFI	$LI_{ij} = 100[(X_{ij}-M_{ij})/(X_{ij}+M_{ij})-S_k(X_{ik}-M_{ik})/S_k(X_{ik}+M_{ik})](X_{ij}+M_{ij})/S_k(X_{ik}+M_{ik})$ *X* and *M* are exports and imports for country *i,* good *j* .	Country is considered to have a comparative advantage (disadvantage) in a given commodity when the balance in relation to GDP exceeds (is less than) the attributed balance, i.e. exceeds (is less than) zero. The comparative advantage neutral point is thus when the net exports marks zero.

Source: own representation of the main theories (6,7,8)

2. RESULTS AND DISCUSSION

We have calculated all the indicators described in Table 2. To analyze the trade patterns and changes in the Balkan States, on the European Union (UE 28) clothing markets during the period of 2000-2015.

As can be seen, the Balassa index is decreasing for all countries in the Balkans. In 2015, countries with the highest RCA index of competitiveness at garment industry on the EU market are Albania (8.07), Republic of Macedonia (5.64) and Turkey (5.04).The largest decreases at the RCA index were recorded in Albania, Republic of Macedonia and Turkey where the value in 2015 was equal with the half value recorded in 2000. The countries that registered a comparative disadvantage in the production of clothing are Montenegro (0.14) and Slovenia (0.41).

According to the index Vollrath which takes into account not only exports but also imports, the same decreasing trend is observed. In 2015, the highest value of the index VRC was registered in Macedonia (2.83) and in Turkey (2.46) followed by Albania (1.74) and Bulgaria (1.69). Slovenia (-0.27) and Montenegro (-1.86) have negative values. This means that these countries have a comparative disadvantage at the garment export on the EU 28 market.

After Lafay index most specialized countries are Macedonia (5.15), Albania (5.12) and Turkey (4.38).

In conclusion, regardless of the indicator used, the most competitive countries in the clothing's trade are Albania, Republic of Macedonia and Turkey while at the opposite pole is Montenegro and Slovenia.

Table 3. Evolution of comparative advantage indicators, reported to EU 28, 2000-2015.

		2000	2001	2002	2003	2004	2005	2006	2007	2008	2009	2010	2011	2012	2013	2014	2015
RCA	Albania	16.05	15.05	15.11	14.50	14.69	14.21	14.05	13.57	13.39	12.48	9.35	8.65	7.86	7.19	6.83	8.07
	Bulgaria	6.30	7.52	8.00	8.43	7.93	6.98	6.04	5.46	4.50	4.52	3.85	3.34	3.20	3.08	3.07	2.88
	Bosnia and Herzegovina				2.05	1.72	1.62	2.28	2.15	2.14	2.18	1.81	1.81	1.82	1.87	1.96	2.12
	Croatia	4.59	4.43	4.39	4.07	3.53	3.07	2.59	2.39	2.21	2.28	2.13	2.25	2.21	2.09	2.82	2.64
	Greece	5.67	5.44	5.62	5.65	5.34	4.39	3.71	3.38	3.12	2.57	2.01	1.73	1.42	1.35	1.27	1.13
	Montenegro							0.05	0.07	0.12	0.21	0.18	0.09	0.15	0.11	0.17	0.14
	Romania	9.71	10.28	9.88	9.78	9.02	7.89	6.83	5.41	4.25	3.52	3.20	3.00	3.06	2.68	2.53	2.37
	Serbia							2.47	2.55	2.60	2.98	2.14	2.12	2.38	2.11	2.06	1.90
	Slovenia	1.92	1.71	1.33	1.14	1.05	1.06	0.92	0.84	0.72	0.63	0.61	0.49	0.45	0.43	0.45	0.41
	Macedonia	10.41	11.61	12.62	12.68	12.81	11.55	10.56	9.44	10.65	10.06	8.67	7.63	7.94	7.44	6.60	5.64
	Turkey	10.19	8.95	9.41	8.93	7.96	7.62	7.03	6.54	5.32	5.28	5.77	5.36	4.96	5.20	5.15	5.04
VRC	Albania	2.11	2.11	2.06	1.98	2.02	2.06	2.01	1.97	2.10	2.36	2.00	1.96	1.86	1.67	1.68	1.74
	Bulgaria	2.00	1.96	1.88	1.91	2.02	2.14	2.14	2.10	2.04	2.02	1.83	1.87	1.85	1.78	1.74	1.69
	Bosnia and Herzegovina				1.26	1.10	0.76	1.13	1.18	1.22	1.30	1.08	1.15	1.09	1.10	1.13	1.22
	Croatia	1.43	1.65	1.84	1.77	1.61	1.44	1.17	0.96	0.93	0.93	0.82	0.79	0.78	0.81	0.68	0.66
	Greece	1.71	1.65	1.94	1.88	1.72	1.41	1.24	1.00	0.89	0.75	0.54	0.47	0.43	0.40	0.34	0.16
	Montenegro							-2.02	-2.08	-1.91	-1.28	-1.52	-2.27	-1.81	-2.24	-1.72	-1.86
	Romania	2.54	2.60	2.55	2.61	2.69	2.63	2.62	2.41	2.08	1.91	1.89	1.82	1.76	1.68	1.64	1.58
	Serbia							1.85	1.62	0.89	1.63	1.51	1.32	1.53	1.38	1.33	1.37
	Slovenia	0.57	0.50	0.34	0.29	0.51	0.56	0.42	0.32	0.09	-0.11	-0.06	-0.24	-0.24	-0.23	-0.24	-0.27
	Macedonia	4.21	3.93	4.10	3.83	3.77	2.81	2.95	2.95	3.17	3.15	3.12	3.19	3.16	3.03	2.98	2.83
	Turkey	4.22	3.94	4.06	3.90	3.65	3.56	3.28	3.04	2.63	2.46	2.46	2.48	2.55	2.54	2.53	2.46
LFI	Albania	9.60	9.07	8.82	8.59	8.71	7.83	7.40	6.94	6.93	7.09	5.48	5.07	4.61	4.33	4.30	5.12
	Bulgaria	5.77	6.97	7.25	7.60	6.88	5.81	4.77	4.17	3.31	3.70	2.75	2.42	2.27	2.20	2.26	2.09
	Bosnia and Herzegovina				0.98	0.71	0.40	1.02	0.97	0.97	1.14	0.73	0.79	0.74	0.80	0.89	1.06
	Croatia	3.25	3.45	3.48	3.08	2.45	1.84	1.22	0.90	0.77	0.82	0.58	0.57	0.57	0.58	0.56	0.42
	Greece	3.77	3.64	3.96	3.69	3.06	2.18	1.58	1.08	0.82	0.49	0.12	0.03	-0.01	-0.06	-0.15	-0.42
	Montenegro							-0.40	-0.44	-0.49	-0.53	-0.62	-0.76	-0.67	-0.81	-0.74	-0.71
	Romania	9.84	10.69	10.27	10.10	8.81	7.19	5.80	4.30	3.13	2.82	2.33	2.13	2.10	1.85	1.80	1.64
	Serbia							1.43	1.39	0.95	1.96	1.19	1.12	1.36	1.22	1.20	1.13
	Slovenia	0.46	0.31	-0.02	-0.09	0.15	0.18	0.02	-0.07	-0.24	-0.51	-0.41	-0.47	-0.41	-0.40	-0.47	-0.48
	Macedonia	11.17	12.94	13.46	13.56	12.77	10.56	9.29	8.21	8.99	9.11	7.38	6.54	6.62	6.41	6.01	5.15
	Turkey	10.30	10.14	10.56	9.87	8.14	7.32	6.27	5.70	4.40	4.77	4.56	4.13	3.93	4.17	4.42	4.38

3. CONCLUSION

The study of the Balkans shows that although most countries have remained competitive, the degree of competitiveness has a negative trend. In order to maintain their competitive advantage, the countries concerned must seek to develop the main factors of competitiveness: labor, capital, but also factors related to production technology, price differences, consumption, and productivity. They also need to capitalize some of the advantages like: local endowments, quality and availability of infrastructure, and proximity to input and product markets.

The main challenges faced by the Balkan states were restructuring of the economic system, changing markets and trade patterns, declining domestic demand for brand less clothing, reducing competitive capacity and economies of scale through outsourcing or subcontracting, and reducing the export base. For a long time, cheap labor has been one of the main strengths of the Balkan clothing industry. Rising wages in this industry, as a result of labor market pressure or government policy, has lead to decreasing competitiveness of these products on the EU market and implicitly to the decrease of exports of garments from these countries. Opening up the labor market for EU citizens, or migrated to better paid sectors, leads to a shortage of workers, especially skilled, which in the long run may lead to lower product quality. Possible effects, to maintain competitiveness, would be pushing the industry into automated technology to reduce human costs, or get higher value-added products. Both variants lead to other competitiveness factors: production technology and capital; factors that are deficient in this area. A possibility of maintaining the competitive advantage is a better strategic positioning of the companies by adopting a diversification strategy. The main opportunities that most of these countries can exploit are related to their geographical position and infrastructure in the garment sector.

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IMPROVEMENT OF LIQUID MOISTURE MANAGEMENT IN PLAITED KNITTED FABRICS

Muhammad Zaman KHAN, Sajid HUSSAIN, Hafiz Faisal SIDDIQUE, Vijay BAHETI, Jiri MILITKY, Musaddaq AZEEM, Azam ALI

Faculty of Textile Engineering, Technical University of Liberec, Studenstska 2, Liberec, Czech Republic

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ABSTRACT

The main purpose of this study is to investigate the effect of knitted fabric structures and material composition on moisture management properties. Fiber type and fabric structure have significant influence on moisture management properties of knitted fabrics. In this article, single jersey, plaited jersey and hybrid plaited jersey knitted fabric samples with different yarn compositions were prepared. Air permeability and liquid moisture management properties including wetting time, max wetted radius, absorption rate, one-way transportation capability and OMMC were evaluated. Plaited jersey and hybrid plaited jersey structures have better moisture management properties than that of single jersey knitted structures. In comparison with plaited jersey and hybrid plaited jersey structures, air permeability of single jersey knitted structures is better.

Keywords: Single jersey, Plaited jersey, Thermo physiological comfort, Moisture management

Corresponding Author: Muhammad Zaman Khan, zamankhan017@yahoo.com

1. INTRODUCTION

Clothing designed for leisure sports is worn not only for aesthetic reasons but also for special control functions of the human body. Clothing comfort includes three main considerations: thermo-physiological, sensorial and psychological comfort (1).

Human thermal comfort depends on a combination of garment, climate, and physical activity. Main factors which affect thermo-physiological comfort are fiber type, yarn properties, fabric composition, fabric structure and chemical finishing (2).

Sweat shirts are widely used in sports, army and personal wears. Mostly knitted sweat shirts are manufactured according to the requirement of the wearer. Sweat shirts like hoodie are used in cold environment.The main function of sweat shirt is to make wearer dry, cool, fresh and comfortable. Mainly cotton fiber is used for manufacturing of sweat shirts. Synthetic fibers are also used for this purpose. Many research works are carried out to study the functional properties of double layered fabric such as heat and moisture transport properties (3).

When human body temperature goes beyond from normal range due to hot and humid weather or heat generated during physical activity due to metabolic activities. In this case human body acts a cooling system and starts sweating. To increase the comfort level clothing must ensure transmission and evaporation of moisture. Sweat must be transferred or removed from the surface of the skin towards the fabric that is next to the skin. After absorbing moisture, fabric should wick out moisture to the atmosphere in order to reduce the humidity (1). There is a general agreement that the transfer of heat, moisture and air through the fabric are major factors for thermal comfort (4).

Sweat should be transported away from the skin in the form of moisture or liquid in order to keep the skin dry. The ability of a garment to transport moisture away from skin to the garments outer surface is called as moisture management (5).

Thermal diffusivity, air and water vapor permeability are influenced by raw material type and knitted fabric structure. Wicking and drying of a fabric is greatly influenced by the selection of raw material and knit structure.

Natural fibers such as cotton having hydrophilic nature have bonding sites for water molecules and water tends to remain in the fibers. Cotton has poor tendency of moisture transportation. Synthetic fibers such as polyester are hydrophobic and have few bonding sites for water molecules. Hence, they tend to remain dry and have good moisture transportation and release properties (6).

Sweat shirts made of 100% cotton fiber absorb lot of moisture but wicking rate is poor that is why it causes irritation and discomfort to the wearer because of getting

wet. Shirt made of 100% polyester has low moisture absorbency and causes irritation in hot conditions.

Double layer fabrics are used to ensure better clothing comfort in sports and active wears. High moisture transfer properties of these fabrics affect their comfort properties and make them more functional. Commonly, a simple double-layer construction is used in which the inner face is made of a synthetic filament yarn that is hydrophobic in nature and has a good capillary action; the outer face is made of a hydrophilic yarn that absorbs the wicked moisture and allows it to evaporate (7).

Most important component of the clothing comfort is thermo-physiological comfort. It is of great significance because of tremendous support to the active leisure. It is also preferred to balance the human body's core and skin temperature at varying environmental conditions and extent to normalize the micro-climate between the wearer's skin and the garment. Numerous factors that influence the thermo-physiological comfort properties of a wearer are heat exchange within clothing, air permeability, transfer and evaporation of moisture. Wetting, wicking and moisture management are critical characteristics that affect thermophysiological comfort of a garment (8).

The transport of liquid within a fabric can take place through the available inter yarn spacing and through the inter fiber space in its constituent element. Such transmission takes place primarily by capillary action. So, the generation of inter and intra yarn spaces in fabric is expected to influence transportation of liquid (9).

The human body produces heat continuously inside his/her body during all his/her activities because of metabolic activities. Due to excessive physical exertion and greater level of heat generated by the body itself, heat transfer through clothing is insufficient to compensate for the body's energy balance. In this case, body begins to sweat and due to evaporation of this sweat, human body feels cooling effect. Human body remains dry in order to avoid discomfort and other skin related diseases (3).

The transfer of moisture through fabrics takes place via two main processes; wicking and absorption. Good wick ability of fabric can help efficient transport of liquid from skin surface to the outer surface of the fabric. Similarly, moisture absorption refers to the uptake of moisture byindividual fibers from the surface of skin. Natural fibers like cotton and regenerated cellulose fibers show much higher absorption abilities in comparison with synthetic fibers (10).

Dryness of body plays vital role to keep it free of perspiration and bacteria that causes variety of problems like blisters and athletic deceases. These problems can be resolved if we are able to develop a garment in which the wearer feels comfortable. In order to develop such type of garment, we will have to emphasis over the bio-physical aspects of the garments in the form of liquid/moisture transportation and permeation of air through the fabric.The sweat transportation in the form of liquid or vapor consequently through the fabric touching the skin causes dryness (11).

Pernick et al., studied the moisture absorption and wicking properties of multi-layer weft knitted fabrics. This multi layering is comprised as a first layer of hydrophobic yarns and a second layer of hydrophilic yarns. These layers being secured together by series of courses forming spacer yarns. The hydrophobic layer comprised polyester yarns while the hydrophilic layer comprised cotton and rayon yarns (12).

Chen et al., designed a new double jersey knitted structure and compared it with 1×1 rib structure and a double jersey structure knitted with different yarns as face and back loops. He analyzed and concluded the significantly better initial water absorption rate and accumulative one-way transport properties as compared to conventional knitted structures. Continued to his work, plant-based design which contains two tucking courses sandwiched between two knitting courses has shown the best results in fabric comfort properties (13).

Supuren et al., has developed double-face knitted fabrics comprised of only two type of fiber incorporated as yarns in four different options like (i.e. cotton-cotton, polypropylene-cotton, cotton-polypropylene, polypropylene-polypropylene) on face and back of the fabric have been compared for moisture management properties. Established results of the fabric samples with cotton as (outer-side) and fabric with polypropylene (inner-side) showed the best moisture management properties (7).

In a previous study., Only effect of fabric physical property and knitting parameter on comfort-related properties of commercial sportswear fabric was investigated (14).

In a study it was observed that slack forms of rib, interlock and single jersey structures have higher transfer wicking ratios as compared to their tight forms. All tight knitted structures had higher contact angles than their slack forms due to higher compactness of the surface. The test results revealed that the parameters of comfort are significantly affected by knitted structure (15).

In a study carried out by Ozdil et al., cotton yarns with different yarn counts (Ne 20, Ne 30, Ne 40) and different twist values (α_e = 3.2, 3.6, 4.0) were knitted as single jersey structure in the same production conditions. The moisture management properties of the fabrics were measured. Overall moisture management capacity (OMMC) values of all the cotton fabrics were found in the same category even the yarn counts and yarn twist coefficients were different (16).

In this study, plated knitted fabrics produced with functional fiber yarns in the back of the knit (close to the body); combined with polypropylene or polyester in the face (outer surface) were tested in terms of their wicking behavior and drying rate capacity. The wicking behavior of fabrics is mainly determined by the effective capillary pore distribution and pathways as well as surface tension. The drying capability is related to the macromolecular structure of the fiber. Viscose Outlast® demonstrated the best wicking ability in both horizontal and vertical wicking, but showed low drying capability. Coolmax® showed a good wicking ability and the best drying capability (17).

Fabric structure seems to be much more important for effective achievement of moisture management properties. Major consideration in the knitted fabric structure is that which layer we are using next-to skin and are mostly more hydrophobic and better wicking yarns which absorb less amount of moisture, and the outer fabric layer of knitted

structure has yarns which pull the moisture outwards and facilitate quicker evaporation. Selection of all these parameters can be important for making a knit structure of best moisture management properties (18).

Foshee et al. has patented his work by developing dual layer knitted fabric. This fabric contains water-absorbent yarns on the outer side and water-wicking yarns on the inner next-to-skin side. Water-absorbent yarns used are cotton, viscose, blend of polyester and cotton with about 85% cotton and 15% polyester. The wicking yarn used is profiled fibers such as Coolmax® polyester (19).

There are numerous studies in fabric liquid management properties that depend on the fiber properties, yarn properties, fabric structure (single jersey, plaited jersey, double faced). But it is first time we developed a blend of samples having single and plaited jersey knit structures. The objective of preparation of single jersey and plaited jersey structures was to compare the liquid transport properties of structures. Development of plaited knit structures with micro denier polyester and trans- dry fabrics is a new work which differentiate liquid transport properties than that of single jersey trans dry fabrics. Fabrics especially use as under garments requires the immediate transport of sweat. But second layer should be absorbent so that the wicked moisture should be absorbed by second layer. But spread ability and drying of liquid moisture is very important phenomena for knitted fabrics specially used as under garments and active wears. That is why we used outer layer as trans- dry fabric because spread ability and dry ability of trans- dry fabrics is better than that of other fabrics prepared by natural and synthetic materials. So development of hybrid structures is a new work which showed better liquid transport results as compared to single jersey structures. Similarly hybrid knit structures showed better moisture transportation results than that of the plaited jersey knit structures that are prepared by natural and synthetic yarns.

This study investigates the comparison of liquid transportation and air permeability of single jersey, plaited jersey and hybrid plaited jersey knit structures.

2. MATERIALS AND METHODS

2.1 Materials

Total eight yarn samples (30/1 Ne) were selected and used in combinations to prepare nine weft knitted fabrics. All samples were produced on circular weft knitting machine (Mayer & Cie, Germany) with machine gauge of 24 and 34" diameter using constant setting values. Testing results of yarns used are given below in Table 1. Out of 9 samples, 3 were single jersey (SJ1, SJ2 and SJ3), 3 were plaited jersey (PJ1, PJ2 and PJ3) fabrics with different blend ratios and 3 were hybrid plaited jersey (HPJ1, HPJ2 and HPJ3) fabrics in which different yarns were used in 1:1 ratio on front side of the fabric. Figure 1 shows the construction of plaited jersey. All fabric specifications are mentioned in Table 2 along with their compositions. Inner layer of the fabric was composed of micro polyester filament yarn 100% (45denier) for only plaited jersey fabrics.

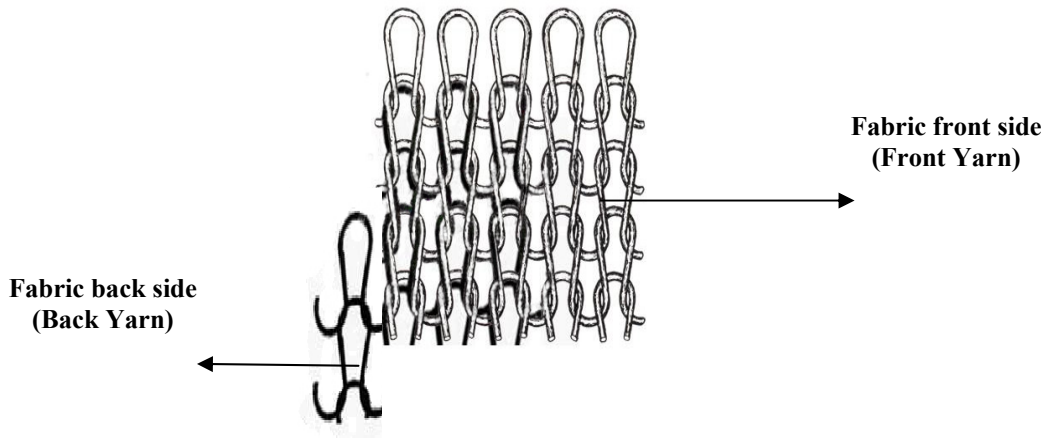

Figure 1. Construction of plaited knitted fabric

Table 1. Test results of yarns used

Type of yarns	Count (Nec)-CV%	Strength (lbs)	CLSP	U %	IPI	Hairiness
30/1 Ne CMB Treated cotton 100%	30/1-0.77	78.3	2485	10.65	147	4.9
30/1 Ne CMB cotton 100%	30/1-0.97	72.1	2157	10.42	97	5.84
30/1 Ne Spun Polyester 100%	30/1-0.93	190	5653			
30/1 Ne CVC 60/40	30/1-0.59	85.6	2533	11.99	430	5.97
30/1 Ne Cotton/ Polyester/ Rayon (50:38:12)	30/1-0.68	82.5	2464	13.36	1185	6.09
30/1 Ne PC 50:50	30/1-1.05	194	5808			
30/1 Ne Cotton/ Coolmax® 60:40	30/1-0.62	87.6	2585	12.05	445	5.99

Table 2. Details of fabric samples and compositions

Sample Code	Front yarn (FY1)	Front yarn (FY2)	Back yarn (BY)	Composition Results
SJ1	30/1 Ne. CMB cotton 100%			100% cotton
SJ2	30/1 Ne. Cotton/ Polyester/ Rayon			50% cotton, 38% polyester, 12% rayon
SJ3	30/1 Ne PC 50:50			Polyester/Cotton 50:50
PJ1	30/1 Ne Cotton /Polyester/Rayon (50:38:12)	……	45 Denier Micro Polyester Filament 100%	Cotton /Polyester /rayon 44: 52:4
PJ2	30/1 Ne Cotton/ Coolmax® (60:40)	……	45 Denier Micro Polyester Filament 100%	Polyester/Cotton 55:45
PJ3	30/1 Ne CMB cotton 100%	……	45 Denier Micro Polyester Filament 100%	CVC 80: 20
HPJ1	30/1 Ne CMB cotton 100%	30/1 Ne Spun Polyester 100%	45 Denier Micro Polyester Filament 100%	PC 60:40
HPJ2	30/1 Ne CMB cotton 100%	30/1 Ne CVC 60/40	45 Denier Micro Polyester Filament 100%	CVC 60:40
HPJ3	30/1 Ne CMB cotton 100%	30/1 Ne CMB Treated cotton (Water repellent)	45 Denier Micro Polyester Filament 100%	CVC special 80:20

Table 3. Test results of knitted fabrics

Sample Code	Mass (g/m^2)	Fabric PH (After bleach)	Loop length (mm)
SJ1	165	6.97	3
SJ2	160	7.15	3.2
SJ3	163	6.93	3.3
PJ1	175	6.99	3
PJ2	173	6.91	3.2
PJ3	176	6.95	3.1
HPJ1	176	7.01	3.2
HPJ2	177	6.98	3
HPJ3	176	7.12	3.1

2.2. Applied Treatment

All of the fabric samples were half bleached to remove the natural extracts. For half bleaching, exhaust dyeing machine (Fong) was used and bleaching was done for 25min at 85°C. Recipe used: Hydrogen peroxide 4g/l, caustic soda 3g/l, stabilizer and wetting agent. After half bleaching samples were dried, conditioned and tested in the standard atmosphere conditions. No chemical or mechanical finish is applied. The pH value of the half bleached fabric was tested to test the acid and alkali contents remained in fabric. The pH value will not make the skin itchy if controlled between weak acid and neutrality. The test method AATCC 81-2006 was used to measure pH of the water extract from half bleached fabric. This test method determines the pH of wet processed textiles. 10 g specimen was cut into small pieces and immersed into boiling distilled water (250 ml) for 10 minutes. Finally the water cooled down to room temperature and pH of the extract was determined using a pH meter. The pH values are given in Table 3.

2.3. Moisture management

To measure moisture and liquid transportation of sportswear, moisture management tester (MMT) was established as shown in Figure 2. This tester is very unique in its construction and it fulfills all the aspects of sweat management for fabric's top and bottom sides (20).

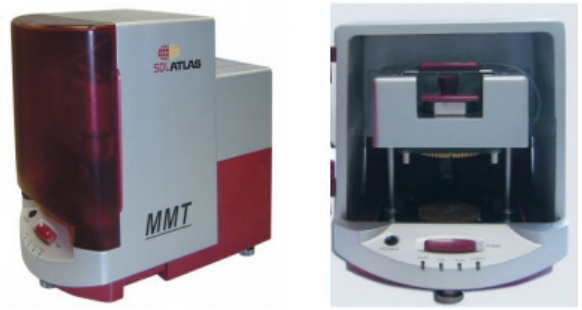

Figure 2. Moisture Management Tester

The liquid management trend is in the form of 'wetting time', 'absorption rate', 'one way transport capability', 'spreading/drying rate' and 'overall moisture management capacity'. Wetting time is actually the time in which tested fabric is wetted. Absorption rate is the speed at which the mean quantity of generated sweat is absorbed during the initial water content in textile material. Spreading speed is the accumulated rate of surface wetness to a maximum radius from the point at which the water droplet falls. The

maximum wetted radius is the greatest water ring radius measured on the surface of fabric. Accumulative one-way transport capability is the difference between the areas of the liquid moisture content curves of a fabric with respect to time (21, 22, 23).

Overall (liquid) moisture management capability (OMMC) is overall ability of the fabric to manage the transport of liquid moisture. OMMC is calculated by using following equation (1).

$$OWTC = C1MARb + C2OWTC + C3SSb \quad (1)$$

Here, C1, C2, and C3 are the weights of the index of the absorption rate.

Moisture management properties of the conditioned samples were measured by moisture management tester (MMT). Moisture management test was performed according to the AATCC Test Method 195-2009. The moisture management tester is designed to measure, sense and record the liquid moisture transport characteristics of the fabric in multiple directions (24). An average of five readings was taken for each sample.

2.4. Air permeability

Textest FX instrument was used to measure air permeability of different knitted samples and measurement was done according to standard ASTM D2986 test method. Measurement was done at the range of 3 and pressure 200 Pa. Principle of this instrument depends on the measurement of air flow passing through the fabric at a certain pressure gradient ΔP.

3. RESULTS AND DISCUSSION

3.1. Moisture management

Wetting time was investigated for all samples having different material composition and knitted structures. Single jersey samples showed almost same wetting time as compared to the plaited jersey samples and some of hybrid plaited jersey samples. It can be seen in Figure 3 that PJ and HPJ samples took less time to absorb water drop except HPJ3 sample. HPJ3 took more time because it has one water repellent front yarn.

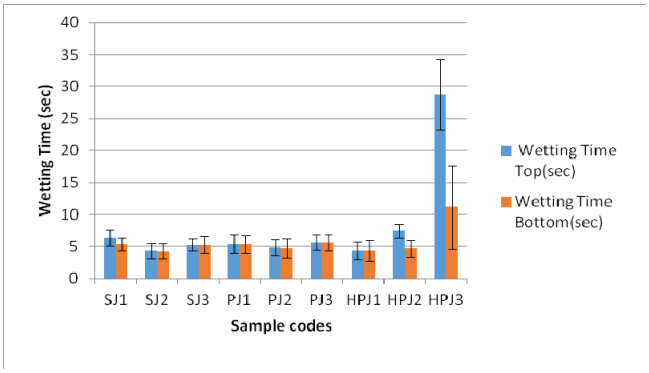

Figure 3. Comparison of wetting time between different knitted structures

All fabric samples' wetted radius has observed within specific time frame. Higher values of wetted radius have observed in PJ and HPJ samples as shown in Figure 4. HPJ2 showed highest wetted radius for top (23mm) and bottom (26mm). The microcirculatory environment imparting the property of higher rate of sweat transportation through the fabric depends on the construction and thickness of fabric. It also causes capillary action and provides a media of prompt spreading of sweat on inner side and outer side of fabric. It can be concluded that samples having micro polyester on inner side of the fabric enhance the moisture transportation by capillary action.

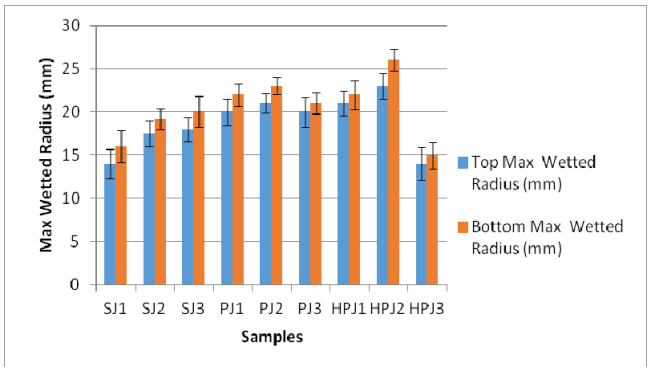

Figure 4. Comparison of wetted radius between different knitted structures

Over all the maximum absorption rate value has been observed at inner side of samples which is in direct contact with skin. Inner side of PJ and HPJ samples are composed of micro polyester. PJ and HPJ samples exhibit high absorption rates as compared to single jersey samples as shown in Figure 5. Absorption rate depends upon the fiber composition of fabric. Synthetic fibers like polyester and polypropylene are used for manufacturing of different variety of fabrics. Natural cotton fibers tend to absorb and retain body moisture, whereas synthetic fibers tend to improve wicking away from the body leading to evaporation of the moisture.

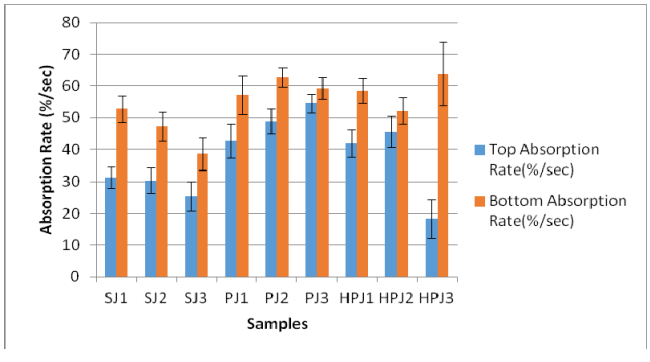

Figure 5. Comparison of absorption rate between different knitted structures

The evaluation of one way transportation capacity value with respect to different type of sample, PJ and HPJ samples exhibit highest value of one way transportation capacity of sweat generated inside the fabric as compared to the single jersey samples as given in Figure 6. HPJ3 had highest value due to the presence of water repellent yarn on the front side of the fabric. This higher side trend in micro polyester fiber containing yarn is due to existence of microcirculatory environment produced inside the yarn construction and fabric as well. This micro circulatory environment is due to implementation of capillary action which may help the sweat to transport from the skin of human body to outer lay while well contributing to provide

maximum comfort and ease of higher sweat release within minimum span of time.

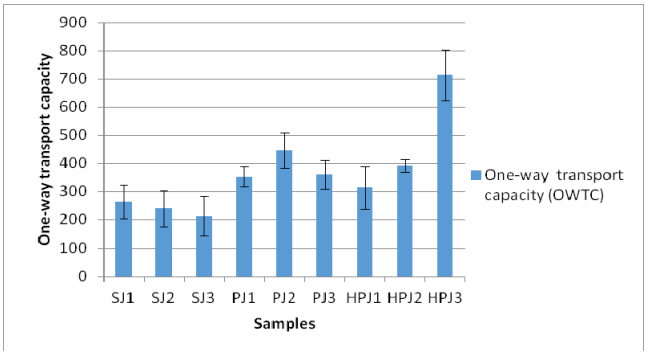

Figure 6. Mean comparison of one-way transportation capacity of samples

Moisture Management phenomena depends on wetting and wicking ability of fabric. We have compared the overall moisture management capacity of all samples as shown in Figure 7. Overall moisture management capacity (OMMC) is actually the quantitative analysis of transportation of sweat generated inside the fabric. Normally it is the combination of one way transportation of liquid, moisture absorption and spreading rate inside the fabric. Out of all samples plaited jersey and hybrid plaited jersey samples shown best results for OMMC. Sample PJ2 has greater overall moisture management capacity than all other samples. But sample HPJ3 has lowest OMMC value which is due to the presence of cotton yarn treated with hydrophobic finish. PJ2 and HPJ2 samples have excellent OMMC values and falls in grade 5 of OMMC grading system. All other samples (SJ1, SJ2, SJ3, PJ1, PJ3 and HPJ1) have very good OMMC values and falls in grade 4 categories except HPJ3 sample which has 3 grading points.

PJ2 sample whose front side is made of 30/1 Ne. 60% cotton, 40% Coolmax yarn and back side made of 45 denier 100% micro polyester exhibits highest moisture management properties among all the samples as shown in Figure 7. Blending has an important role in moisture-related comfort properties of clothing. Plaited jersey samples showed good moisture management properties due to the presence of hydrophobic inner side and hydrophilic outer side, inner side helps to transport sweat from skin to outer side of the fabric and enhance the moisture transportation due to capillary action. Presence of hydrophobic fibers such as polyester helps to increase the wicking property of the fabric.

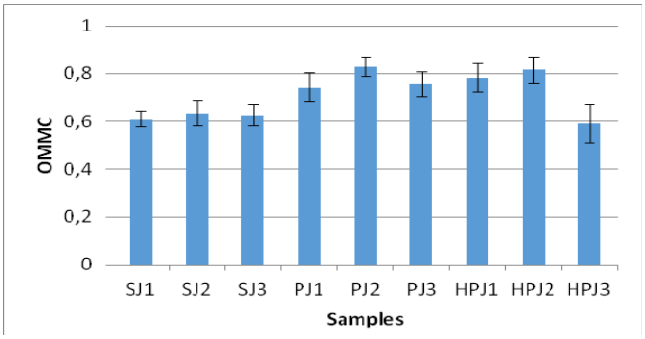

Figure 7. Mean comparison of Overall moisture management capacity (OMMC) of samples

3.2. Relation of OMMC to Air permeability

Figure 8 shows the relation between OMMC and AP. It can be seen that as AP is increasing the OMMC is decreasing. Sample PJ1, PJ2, PJ3, HPJ1 and HPJ2 showing high OMMC values but low AP values. Moreover, single jersey samples exhibited high AP than plaited and hybrid jersey fabric. It can be seen that OMMC is increasing with decrease in AP. Thus, it can be concluded that fabric structures have a great effect on air permeability. Permeability and porosity is strongly related to each other. If a fabric has very high porosity so it is more permeable (1, 25, 26). It can be assumed that there is no significant relation between OMMC and air permeability.

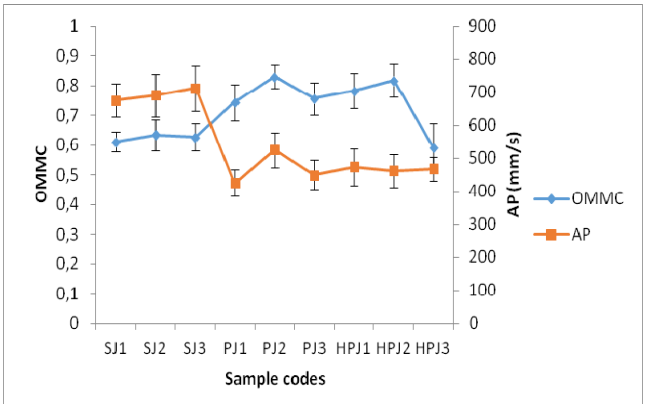

Figure 8. Comparison between OMMC and air permeability of samples

4. CONCLUSIONS

This study mainly focuses on the effect of structure and material composition of single and plaited jersey fabrics on the moisture management and air permeability properties. Double face structure is used with different composition of yarn on front side of the fabric and only one yarn is used on back side of the fabric which is 45 denier 100% micro polyester. PJ2 sample whose front side is made of 30/1 Ne. 60% cotton, 40% Coolmax® yarn and back side made of 45denier 100% micro polyester exhibits highest moisture management properties among all the samples. Sample HPJ2 is also in 'excellent' category in term of moisture manage properties. PJ2 and HPJ2 also have good air permeability. Both fabrics can be used as high performance active wear. A double-face structure with micro polyester yarn for the inner surface and cotton/coolmax® blend yarn for the outer surface; can easily transfers the generated sweat while keeping a dry feeling. So it can be recommended for summer, active and sportswear, due to the high moisture management property and high level of comfort in the wet state.

Acknowledgement

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(REFEREED RESEARCH)

THE PROPERTIES OF STIFFNESS AND ABSORBENCY IN KNIT TOWEL FABRICS

ÖRME HAVLU KUMAŞLARDA SERTLİK VE EMİCİLİK ÖZELLİKLERİ

Seval UYANIK[1], Belkıs ZERVENT ÜNAL[2]

1Gaziantep University, Technical Sciences Vocational School, Gaziantep, Turkey
[2]Çukurova University, Textile Engineering Department, Adana, Turkey

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ABSTRACT

This paper examines the effects of fabric structural parameters such as pile yarn count (Ne 30, Ne 26 and Ne 24), ground yarn count (90 Td and 70 Td), and pile loop height (2.2 mm, 2.5 mm and 2.8 mm) on the properties of stiffness and absorbency in the pile loop and the cut-pile loop knit fabrics. For this aim, thirty samples were produced and then the absorbency and softness values of these samples were tested with standard test methods. Consequently, it was seen that sinker height and pile cut process have been effective on hydrophilicity and softness.

Keywords: Pile loop knit fabric, cut-pile loop knit fabric, fabric stiffness, water absorbency, pile height, yarn number.

ÖZET

Bu makale havlu ve kadife örme kumaşlarda sertlik ve emicilik özellikleri üzerine hav ipliği numarası (Ne 30, Ne 26 ve Ne 24), zemin ipliği numarası, (90 denye ve 70 denye) ve hav yüksekliği (2.2 mm, 2.5 mm ve 2.8 mm) kumaş yapısal parametrelerinin etkisini incelemektedir. Bu amaçla otuz numune üretilmiştir ve sonra ilgili standartlar aracılığıyla bu numunelerin emicilik ve yumuşaklık değerleri test edilmiştir. Sonuç olarak, platin yüksekliği ve hav kesme işleminin hidrofilite ve yumuşaklık üzerinde etkili olduğu görülmüştür.

Anahtar Kelimeler: Havlu örme kumaş, kadife örme kumaş, kumaş sertliği, su emiciliği, hav yüksekliği, iplik numarası.

Corresponding Author: Seval Uyanık, uyanik@gantep.edu.tr

INTRODUCTION

Pile loop knit fabrics are widely used by the apparel industry for sportswear and bathrobes due to their good comfort, touch, and aesthetic properties. Plush fabrics or terry fabrics are obtained by knitting two yarns at the same feeder; a ground yarn knitting plain jersey structure that visible at the fabric front side and a plush yarn knitting plain jersey forming plush loop that visible at the fabric back side in pile and plush structures the pile and plush is clearly distinguishable from the base. Pile is considered to stand out at right-angles to the base, whereas plush lies at less of an angle from the base surface (1). The elongated sinker loops are formed over a higher knock-over surface than the normal-length ground sinker loops with which they are plated. The sinker loops show as a pile between the wales on the technical back of the fabric. Cut-pile loop is achieved during finishing; by cropping or shearing the sinker loops in both directions. This leaves the individual fibers exposed as a soft cut-pile loopy surface whilst the ground loops remain intact (2). On the sinker top latch needle machine, the ground yarn is fed into the sinker throat and the sinker is then advanced so that the pile loop yarn fed at a higher level (Figure 1) is drawn over the sinker nib. A range of pile loop heights from 2 to 4 mm is possible using different heights of sinker (2).

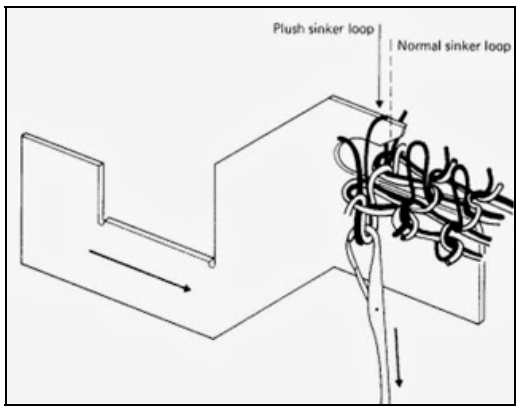

Figure 1. Pile loop formation (2)

As shown in Figure 2, terry (pile loop) fabric can also be knitted on a single knit machine. The face of the fabric is jersey and the terry loops of yarn appear on the technical back of the unfinished fabric. Therefore the fabric is turned inside out for finishing. During finishing, the tops of the loops are sheared, converting a knit terry into terry velour. This produces a soft plush surface on the fabric (3).

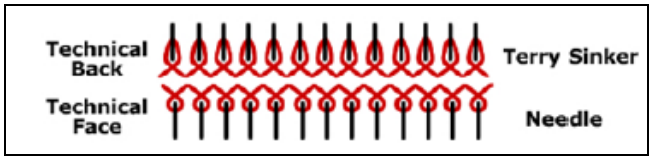

Figure 2. Terry fabric needle diagram (4)

Many studies have reported that the mechanical properties of knitted fabrics vary according to knit structures, fibers, yarns, and densities, which in turn affect the knit's hand significantly. Although there are many studies related to the properties of single jersey and its derivatives, a few studies are available about pile loop knit fabrics in the literature.

Knapton et al. (5-7) reported that the dimensional stability and knit performance of the fabrics are influenced by components such as knit structure, stitch length, and cover factor.

Anand and Lawton (8-9) studied pile-loop knit fabric and presented several empirical models to predict dimensional parameters such as course density, wale density, and stitch density. They reported that the dimensional parameters of pile loop knit fabrics are largely controlled by the stitch length in the ground structure and the state of relaxation. In another study, the same researchers (10) investigated the performance of several knit fabrics, such as pile loop knit, 1x1 knit/miss, and 1x1 cross-inlay, when used as dust filters.

Choi and Ashdown (11) focused on the mechanical properties of weft knits for outerwear as a function of knit structure and density and the relationships between hand, structure, and density in their studies. They reported that tensile properties, stiffness and fullness increase but, compression values, softness and smoothness decrease as knit density increases.

Kim et al. (12) analysed the effect of chemical splitting on the mechanical properties and water absorption of a split-type nylon/polyester microfiber pile knit under various alkaline hydrolysis treatment conditions. They found that hydrolysis parameters and sodium hydroxide concentration affected mass loss of hydrolysed pile knits and their mechanical properties.

Ucar and Canbaz Karakas (13) investigated some physical properties of pile loop knit fabrics. In this study, they showed that the effects of pile type, fiber type, fabric tightness, and relaxation on the physical properties of pile-loop knit fabrics are important. They determined that cut-pile fabric shows more dimensional changes and less spirality than pile-loop fabric. In addition to this, the ground-face fabric has more drapability than the pile-face fabric. Also, there is no significant relationship between pile type and drape coefficient.

Uyanik et al. (14) examined that the effect of fabric structural parameters on dimensional and aesthetic properties in pile loop and cut-pile loop knit fabrics. They found that the higher pile height increases fabric shrinkage, reduces spirality in pile loop fabrics, and increases spirality in cut-pile loop fabrics. In pile loop fabrics, the thicker pile yarn increases shrinkage in wale direction and increases spirality. Ground yarn number is not effective on dimensional stability and spirality.

Uyanik (15) investigated the performance properties such as abrasion resistance, bursting strength, and air permeability of the pile loop and cut-pile loop fabrics. She determined that the bursting strength increases with thicker ground yarn, and the thicker pile yarn reduces the air permeability. Pile loop knit fabrics have higher abrasion resistance and lower air permeability in comparison to cut-pile loop knit fabrics. There is not much difference between the bursting strength of pile loop and cut-pile loop knit fabrics if the linear density of ground and pile yarn is the same.

As seen in previous works, there are no studies on the absorbency properties of pile loop knit fabrics or cut-pile loop knit fabrics. In addition to this, the studies investigating the effects of all factors together, which are pile yarn number, ground yarn number, pile height and pile type, on these fabrics are not found.

In this study taking into account the aforementioned circumstances, it was aimed to investigate the effects of yarn numbers and pile heights on the properties of stiffness and absorbency in pile loop and cut-pile loop knit fabrics. So it is thought that the study in these respects will contribute to the literature.

MATERIALS AND METHODS

Material

In this research, eighteen pile loop knit fabrics and twelve cut-pile loop knit fabrics were produced by using 100% carded cotton ring spun pile yarns and 100% polyester filament ground yarns. The pile yarns are three yarn numbers as Ne 30, Ne 26, Ne 24 and the ground yarns are two yarn numbers as 90 denier and 70 denier. Pile yarn properties were given in Table 1.

Method

Pile loop knit fabrics were produced by using three different sinker heights (2.2 mm, 2.5 mm and 2.8 mm). The machine properties were given in Table 2.

Table 1. Pile yarn properties

Yarn number, Ne	Breaking strength, rkm	Breaking elongation, %	Twist, tpm
30	15.4	4.6	820
26	16.9	4.6	768
24	18.3	5.0	733

Table 2. Knitting machine specifications

Sinker height, mm	2.2	2.5-2.8
Machine type	Orizio-JSVRN	Keumyong-KM-3SV
Gauge	20	20
Diameter, inch	30	30
Feed number	44	44
Needles	1856	1896
Machine speed, rpm	20	20

Produced pile loop knit fabrics were dyed under the same conditions. Finishing process includes the basic stages which are kiering, dyeing, and drying processes, and the auxiliary stages which are washing and neutralization processes. In this phase, the pile loops formed by cotton yarns were dyed while the ground loops formed by polyester yarns were not dyed. Dyeing process was made pad-batch method which is semi-continuous method, and in terms of dye-stuff, hot brand reactive dye-stuff was used due to dyeing in high temperatures. Finishing process machines and stages were shown in Tables 3-4, respectively. Hence, the process diagrams of kiering and dyeing were displayed in Figure 3 and Figure 4, respectively. Then, dyed pile loop fabrics with 2.5 mm and 2.8 mm were sheared to make cut-pile loop fabric, on the other hand the pile loop fabrics with 2.2 mm were not sheared owing to this pile height value was too short to cut. In the last phase of finishing processes, the relaxation process was made for pile loop fabrics and cut-pile loop fabrics to give dimensional stability.

Table 3. Finishing machine specifications

Process	**Machine type**
Pre-treatment	Erbatech
Dyeing	Erbatech
Drying	Elmego Tumbler
Shearing	Biancalani
Drying for relaxation	Brückner

Table 4. Finishing process

Processes		**Process Conditions**
Pre treatment	Kiering	95 °C, 45'
	Washing	30 °C, 10'
	Neutralization	35 °C, 15', pH 7
Dyeing	Reactive dyeing	60 °C, 60'
	Washing	30 °C, 10'
	Neutralization	35 °C, 15', pH 7
	Washing with soap	90 °C, 30'
	Washing with soap	80 °C, 30'
	Washing	60 °C, 10'
Drying		140 °C, 14 m/min
Shearing		15 m/min

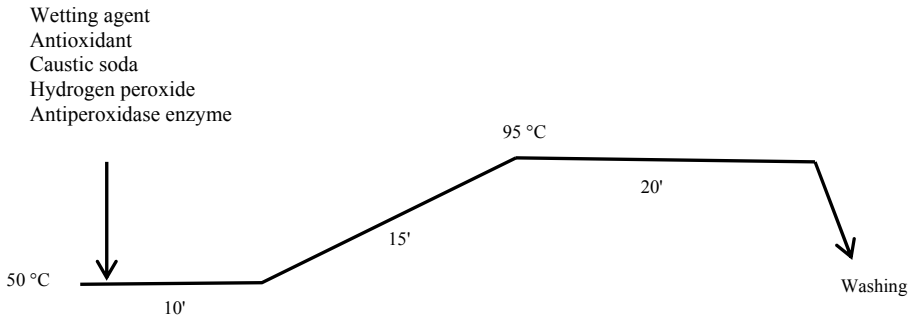

Figure 3. Kiering process diagram

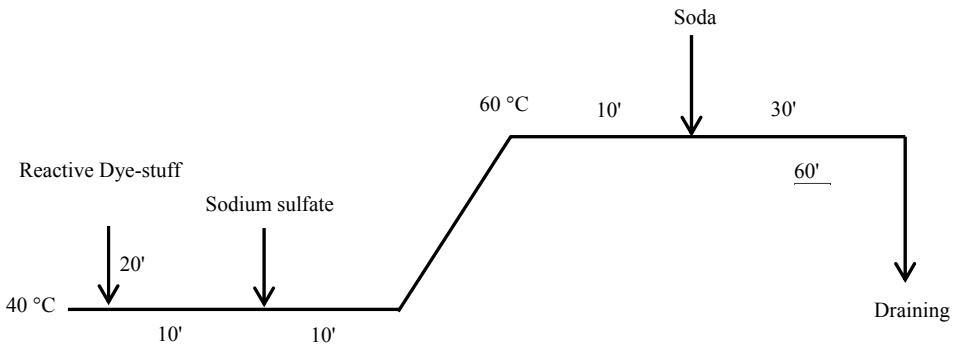

Figure 4. Dyeing process diagram

All the samples were conditioned in accordance with the standard ASTM D1776-08 before testing. The physical properties i.e. course density (cpc), wale density (wpc), stitch density (cpc x wpc), pile/ground ratio, weight, and thickness were measured according to standards EN 14971, TS 629:2015, EN 12127, and EN ISO 5084, respectively. Stiffness and water absorption tests were applied for all fabrics in accordance with ASTM D4032-94 and TS 866 standards. MANOVA analysis was made in order to determine the relationships between variables and the significance of each factor's contribution. For this aim the statistical software package SPSS 21.0 was used to interpret the experimental data. All the test results were assessed at significance levels p ≤ 0, 05 and p ≤ 0, 01.

RESULTS AND DISCUSSION

The properties of pile loop and cut-pile loop knit fabrics were given in Table 5. To identify the samples, the pile loop knit fabrics are coded as P while the cut-pile loop knit fabrics are coded as C. Sinker height and pile height were accepted as the same expressions since the pile height is determined according to the sinker height in the pile knit fabrics. So, sinker height term was used on the following parts.

Structural Properties

According to Table 5, it was seen that cource density (cpc), wale density (wpc), stitch density (number of stitches/cm^2) and pile/ground ratio values of the samples produced in the same machine were same or very close to each other, because machine settings were not changed during production. Fabric weight and thickness values of the samples varied depending on yarn number and sinker height. As expected, usage of thicker yarn and higher sinker height increased the weight and thickness values of the samples. All cut-pile loop samples were expectedly lighter and thinner than pile loop samples because of shearing the pile loop of fabrics. As a result of this, cut-pile loop samples lost weight approximately 19% to 28% and these samples were thinner as ratio of 14% to 30% in comparison to pile loop samples.

Stiffness

When Figure 5 is examined, it can be usually said that pile loop fabrics have higher stiffness values than cut-pile loop fabrics. The samples with 2.8 sinker have higher stiffness values for both pile loop and cut-pile loop fabrics. In the pile loop fabrics, samples with 2.2 sinker are stiffer than samples with 2.5 sinker. There is an increase trend for stiffness by decreasing pile yarn number. In other words, thicker pile yarns increase stiffness values except some samples. Samples containing 90 Td ground yarn have higher stiffness values than samples containing 70 Td ground yarn in pile loop fabrics whereas the stiffness values are close for both of them in cut-pile loop fabrics. Hence, the pile loop samples with 2.8 sinker have higher stiffness than those of the cut-pile loop samples considering the same ground yarn number. The sample of which pile yarn number is Ne 24, ground yarn number is 90 Td, sinker height is 2.8 has the highest stiffness value (the stiffest sample). It was determined that the softest sample is sample code P1 (ground yarn 90 Td, pile yarn Ne 30 and sinker height 2.2).

Table 5. The properties of the pile loop and cut-pile loop samples

Samples	Fabric type	Yarn number		Sinker height (mm)	cpc	wpc	Stitch density (cpc x wpc)	Pile/ground ratio	Weight (g/m²)	Thickness (mm)	Stiffness (kgf)	Water absorbency (sec)
		Ground (Td)	Pile (Ne)									
P1	pile	90	30	2.2	12.5	9	112.5	2.23	214.35	1.58	0.040	8.64
P2	pile	90	30	2.5	12.5	9	112.5	2.53	230.87	1.85	0.075	15.6
P3	pile	90	30	2.8	14	10	140	2.62	259.76	1.94	0.085	18.7
P4	pile	90	26	2.2	12.5	9	112.5	2.23	250.19	1.75	0.065	7.2
P5	pile	90	26	2.5	13	9	117	2.47	259.34	1.83	0.075	8.9
P6	pile	90	26	2.8	15	9.5	142.5	2.69	319.22	2.18	0.100	7.2
P7	pile	90	24	2.2	12.5	9	112.5	2.22	268.48	1.85	0.100	8.6
P8	pile	90	24	2.5	14	9	126	2.40	286.57	1.88	0.065	7.9
P9	pile	90	24	2.8	15.5	9.5	147	2.68	331.50	2.16	0.130	7.9
P10	pile	70	30	2.2	12	9	108	2.21	192.68	1.69	0.075	8.2
P11	pile	70	30	2.5	13	10	130	2.48	236.73	1.85	0.045	7.9
P12	pile	70	30	2.8	14	9.5	133	2.62	242.99	1.75	0.055	9.8
P13	pile	70	26	2.2	13	9	117	2.24	240.05	1.65	0.075	3.7
P14	pile	70	26	2.5	12.5	9.5	119	2.40	240.76	1.75	0.060	6.0
P15	pile	70	26	2.8	14	9.5	133	2.59	279.77	2.07	0.080	11.0
P16	pile	70	24	2.2	12.5	9	112.5	2.23	254.82	1.69	0.075	4.4
P17	pile	70	24	2.5	12.5	9	112.5	2.41	264.33	1.82	0.065	10.0
P18	pile	70	24	2.8	14.5	10	145	2.70	320.24	2.01	0.095	9.8
C1	cut-pile	90	30	2.5	13	9	117	-	171.22	1.29	0.045	12.4
C2	cut-pile	90	30	2.8	15	9.5	142.5	-	210.16	1.59	0.070	13.9
C3	cut-pile	90	26	2.5	13	9.5	123.5	-	203.07	1.41	0.065	7.2
C4	cut-pile	90	26	2.8	15.5	10	155	-	236.46	1.65	0.070	5.2
C5	cut-pile	90	24	2.5	13	9	117	-	204.37	1.40	0.070	5.7
C6	cut-pile	90	24	2.8	15	9	135	-	250.06	1.73	0.085	6.5
C7	cut-pile	70	30	2.5	13	9	117	-	168.92	1.31	0.055	23.8
C8	cut-pile	70	30	2.8	14	9.5	133	-	185.82	1.51	0.080	11.5
C9	cut-pile	70	26	2.5	12	9.5	112.5	-	185.89	1.32	0.060	8.7
C10	cut-pile	70	26	2.8	14.5	9.5	138	-	221.58	1.62	0.065	5.5
C11	cut-pile	70	24	2.5	12.5	9	112.5	-	190.05	1.35	0.070	7.7
C12	cut-pile	70	24	2.8	14	9.5	133	-	239.95	1.71	0.080	5.3

This part was discussed under four sub-headings as structural properties, stiffness, water absorbency, and statistical analyses.

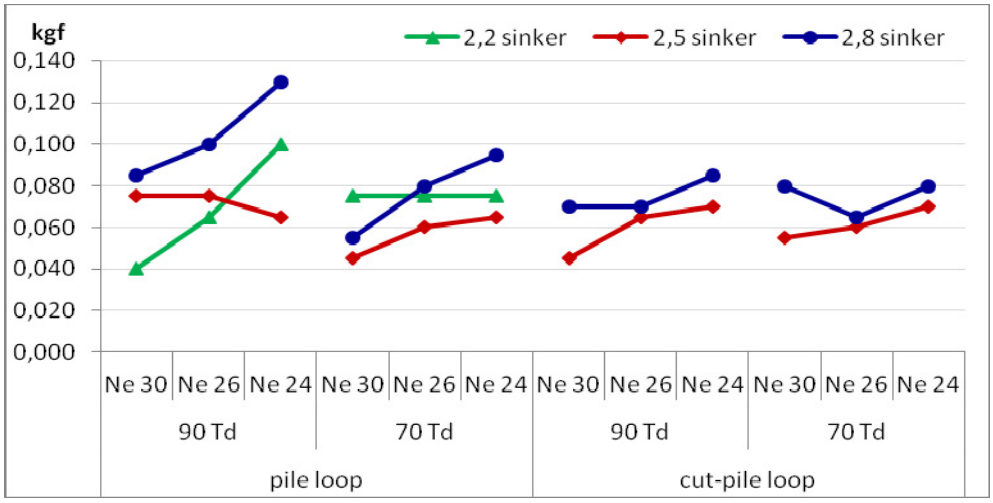

Figure 5. Stiffness

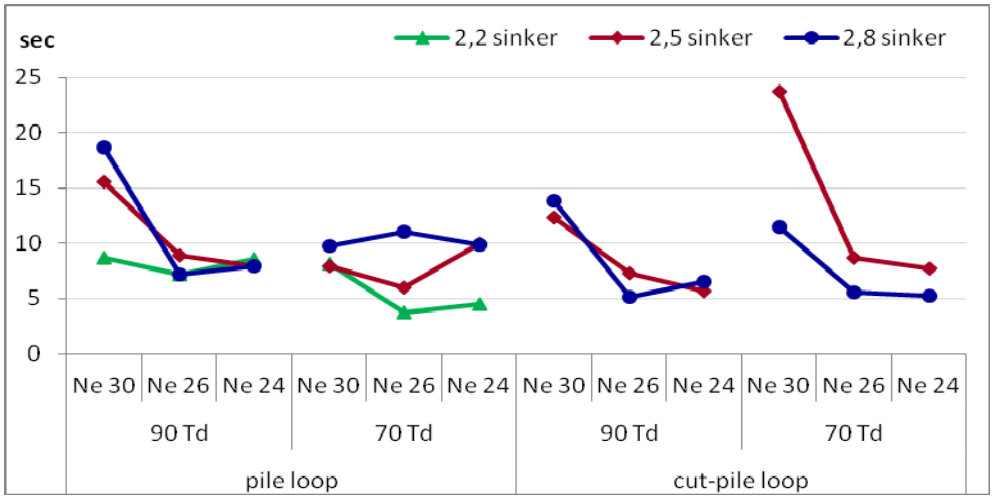

Figure 6. Water absorbency

Water absorbency

The test results and Figure 6 show that the sample of which pile yarn number is Ne 30, ground yarn number is 70 Td, sinker height is 2.5 has the lowest water absorbency. In addition to this, all samples containing Ne 30 pile yarn at different sinker height and ground yarn number have mostly lower water absorbency than the other samples. On the other hand, water absorbency values are very close for both of the samples having Ne 26 pile yarn and Ne 24 pile yarn. In the pile loop fabrics, it is seen that increasing sinker height reduces water absorbency whereas it increases water absorbency in the cut-pile loop fabrics. It is clear that pile yarn is more effective on water absorbency than ground yarn.

Statistical Analyses

MANOVA test results were displayed in Table 6. According to MANOVA results pile yarn has significant effects on stiffness and water absorbency whereas sinker height has significant effect on stitch density and stiffness. Furthermore, ground yarn is not effective factor on all fabric properties.

Table 6. MANOVA test results

Source	Dependent variable	F	Sig.
Ground yarn	Stitch density	1.922	0.191
	Weight	0.687	0.423
	Thickness	0.276	0.609
	Stiffness	0.672	0.428
	Absorbency	0.367	0.556
Pile yarn	Stitch density	0.520	0.607
	Weight	2.622	0.114
	Thickness	0.376	0.695
	Stiffness	**5.123**	**0.025**
	Absorbency	**5.477**	**0.020**
Sinker (pile) height	Stitch density	**59.517**	**0.000**
	Weight	1.869	0.196
	Thickness	1.737	0.218
	Stiffness	**5.595**	**0.019**
	Absorbency	1.507	0.261

Conclusion

Selected experimental and statistical results are summarized below.

- The pile cut process was caused the weight loss and thickness loss in important ratio. So, the raw material cost of the cut- pile loop towels is higher than that of the pile loop towels. The reason for this is the cutting of the ends of the pile.
- In general, the cut-pile loop samples were softer than the other. This may arise from the fact that when the piles are cut off, the yarn twist in parallel with the yarn ends are opened, and so the surface of towel becomes softer.
- According to results, the sinker height is more effective than the yarn number in softness values. As the sinker height increased, the pile loop and cut-pile loop towels hardened.
- Like softness, the water absorbency decreased with increase the sinker height. Especially, the lowest absorbency was obtained with the thin yarn (Ne 30) and high sinker height (2.8) for both the pile and cut-pile loop towels. This situation may be welded from falling easily upon each other of the piles obtained with these yarns.
- As the result of statistical analysis, it was seen that the relationships between stiffness and pile yarn number, stiffness and sinker height, absorbency and pile yarn number were significant statistically.

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(REFEREED RESEARCH)

PROBIOTIC PRINTED PET FABRICS FOR BIOCONTROL IN HOSPITAL TEXTILES

HASTANE TEKSTİLİNDE BİYOLOJİK KONTROL İÇİN PROBİYOTİK BASKILI PET KUMAŞLAR

Kim-Laura NİEHAUS, Aysin DURAL-EREM, Vincent NİERSTRASZ

Textile Materials Technology, Department of Textile Technology, Faculty of Textiles Engineering and Business, University of Borås, SE-50190 Borås, Sweden

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ABSTRACT

Over time, hospital-acquired infections have become one of the main threats to the safety of hospitalized patients. The proliferation of nosocomial pathogens is often reported in connection with hospital textiles. In the present study, probiotic agents were printed on a polyester fabric by means of screen-printing. Afterwards, the viability of the probiotics after application process was determined. The applicability of the obtained fabric was evaluated on the basis of contact angle measurements, abrasion resistance and washing tests which showed the effect on the viability of the probiotics after repeated laundering. SEM images revealed the quality of the printing process. Major findings include that it is possible to obtain probiotic printed fabrics and probiotics were able to survive after the printed on fabrics.

Keywords: Probiotics, Functional printing, Biocontrol, Viability tests, Hospital textiles

ÖZET

Zaman içerisinde, hastane kaynaklı enfeksiyonlar hastaneye yatırılan hastaların güvenliğine yönelik ana tehditlerden biri haline gelmiştir. Nozokomiyal patojenlerin çoğalması hastane tekstiliyle bağlantılı olduğu sıklıkla bildirilmektedir. Bu çalışmada, probiyotik ajanlar, bir polyester kumaş üzerine tarama baskı yöntemiyle basılmıştır. Ardından, uygulama sonrası probiyotiklerin canlılığı belirlenmiştir. Elde edilen kumaşların uygulanabilirliği, kontak açısı ölçümleri, aşınma direnci ve tekrarlanan yıkamalar sonrası probiyotiklerin canlılığı üzerindeki etkisini gösteren yıkama testleri temel alınarak değerlendirilmiştir. SEM görüntüleri baskı sürecinin kalitesini ortaya koymuştur. Temel bulgular, probiyotik baskılı kumaşların elde edilmesinin mümkün olduğunu ve probiyotiklerin kumaşlara basıldıktan sonra canlılıklarını koruyabildiklerini göstermiştir.

Anahtar Kelimeler: Probiyotikler, Fonksiyonel baskı, Biyolojik kontrol, canlılık testleri, Hastane tekstilleri

Corresponding Author: Aysin Dural Erem, aysin.erem@gmail.com

INTRODUCTION

Hospital-acquired infections (HAI) are a major cause of morbidity and mortality among the hospitalized patient (1). These infections are caused by the common nosocomial pathogens (2). Since textile materials such as uniforms, linens and patient apparels are common in healthcare facilities, the proliferation of nosocomial pathogens is often reported in connection with them. Contaminated textiles are an excellent substrate for bacterial growth under appropriate moisture and temperature conditions (3). When they are heavily contaminated, patients and the hospital staff are at a higher risk of getting infected and by this contribute to the distribution of HAI (1, 4). In order to prevent the growth of pathogens and to protect the user from undesirable infections, antimicrobials like silver, triclosan or QAC are introduced into different textile applications (5, 6). Ideal antimicrobial agents should be effective against microorganisms whilst being nontoxic and do not damage the skin flora and cause allergy, skin irritations (7). However, most of these agents work according to a leaching mechanism. This leaching, related to the amount, causes health and environmental problems (8). Sustainable alternatives, based on biological substrates such as enzymes and probiotics receive attention. The use of them as an antimicrobial agent minimizes the amount of hazardous chemicals. Probiotics may defined as live microorganisms generally bacteria or yeasts, which when

locally applied in sufficient numbers confer one or more specified demonstrated health benefits for the host (9). There is a large range of microorganisms with probiotic properties but the most common belong to lactic acid bacteria, *Bacillus* bacteria or yeast, most of them have an antimicrobial or antagonistic ability to inhibit pathogenic bacteria. The antagonistic capability of probiotics against other bacteria can be caused by the competitive exclusion and the production of

organic acids, such as lactic acid, which lead to a reduction in the pH level and thereby provide an unfavourable ground for pathogens (10, 11). In addition, they exhibit antimicrobial activity due to the production of antimicrobial substances (12). When the fabrics are treated with probiotics, they become a reservoir for probiotics and may provide antagonistic/antimicrobial activity against pathogens. The use of probiotic containing products in clinical settings is not unfamiliar in combat of HAI. Vandini and colleagues in 2014, revealed that microbial cleaning with different strains of *Bacillus* spores as part of cleaning products reduces the number of infection related pathogens (13). A subsequent study done by Caselli et al. (2016) investigated the impact of these microbial based cleaning products containing *Bacillus* strains on the reduction of antibiotic resistant bacteria strains. It became evident that the cleaners were not only effective in counteracting the growth of several pathogens; they further did not cause any drug-resistant pathogen population but rather lowered the already existing resistances (14). This research aims to develop strategy to incorporate probiotics in fabrics via traditional textile treatment such as screen printing to inhibit pathogens.

MATERIAL AND METHOD

Material

The printing paste was composed of Tubifast AS 5087 FF binder, Tubivis DL 600 thickener, and Tubiassist Fix 157W cross-linker was provided by CHT R. Beitlich GmbH. The ready-made pigment paste Violett 5 BC was purchased from Zenit AB. The probiotic finishing agent Tana®Biotic DC was kindly provided by Tanatex Chemicals. The exact formula of Tana®Biotic DC is unknown as its a commercial product. The textile substrate was a polyester woven fabric supplied from FOV Fabric AB. Fabric specifications were summarized as weight 146g/m, weft 40 threads/cm, warp 55 threads/cm, carbon yarn density 1/23picks and 1/25ends with 2/2 twill weave.

Method

The thickener was dissolved in water using a plain stirrer. Then the other components were added in thickener solution and stirred for 30min at 400rpm. The ingredients of the different pastes are shown in Table1.

The printing paste was homogenized and applied to the PET fabric using a printing screen with 43-threads/cm mesh. The total number of passes was six. After application, the fabric was dried at 80°C for 15min then cured at 150°C for 5min.

The wettability of the fabrics was evaluated using Theta Optical Tensiometer (Scientific/ Biolin Holding AB). The test was conducted by a water droplet size of 1 or 3μL and the angles of the drops were measured after 0.5sec. Samples were tested for abrasion in a Cromocol, Martindale 2000 abrasion tester according to EN ISO 12947-2AC:2006 standard. Evaluation of the damage and breakage of fibres and searching for pilling will be made at 1000, 2000, 5000, 10000, and 15000 rubs with a visual analyse. The morphology of the fabric surfaces was determined using the environmental scanning electron microscope (ESEM) Quanta 250 FEG (FEI, USA) with 10 kV acceleration voltages.

Table1. Ingredient of the printing pastes

	P1	P2	P3
Binder (g)	6	6	6
Thickener (g)	1.7	1.6	1.5
Cross-linker (g)	-	1	1
Pigment (g)	2	2	2
Probiotic agent(mL)	25	25	2.5
Water (g)	65.3	64.4	87
pH of the paste	6-7	6-7	6-7
Viscosity (Pa.s)	993	919	2270

Viability test for probiotics

The viability of spores attached to the fabrics was evaluated in agar plate test method, which is used to monitor and culture of microorganisms. Prior to the test, the samples (5x5cm) were heated at 75°C for 1h to ensure that they are free from any bacterial contamination. Then the samples put in tryptic soy agar (TSA) plate and 4.5mL of 1% Triphenyl tetrazolium chloride (TTC) solution to fully cover the samples. The agar plates were incubated at 30°C for 48h. After incubation, the growth of bacteria was evaluated. In order to determine the durability of probiotics on fabrics, the fastness to washing test was performed using Wascator FOM 71 MP (Electrolux) according to ISO 6330:2012. The viability of the probiotics was evaluated after 3 and 5 repeated washes.

RESULTS & DISCUSSION

Screen-printing was used an application method as the probiotic spores were entrapped within the paste and easily applied on the polyester fabric. Firstly, the viability of probiotics on a fabric after application was evaluated with agar plate method and results are summarized in Table 2, where the red points represent colonies of beneficial bacteria. As the colonization of the probiotics was so dense, the exact counts could not obtained and estimated counts were given in the results.

According to these results, all samples exhibited a certain amount of growth more than 10000 CFU/surface. Recent studies mainly focused on the encapsulation or incorporation of probiotics into the textile fibre during electro spinning or melt spinning processes and obtained high viability results after the applications (15-17). Therefore, process conditions of screen printing are a promising approach for the application of probiotics on the fabric. Since the dimensions of probiotics cells are approximately 1-2 μm in length and 0.5-1μm in breadth (18), they can be easily embedded in the viscous paste and applied. For the further evaluation of the viability of probiotics, the fastness to washing of the printed parts of the fabrics and

the viability of the probiotics after 5 and 3 washes were evaluated (Table 4). Fastness tests showed the viability of the probiotics may decrease related to the washing cycles. While it is possible to observe some growth on all samples after three washes, the most of the samples couldn't exhibit enough growth of probiotics after five washes.

Table 2. The viability results

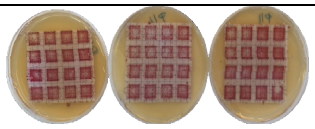
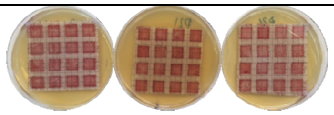
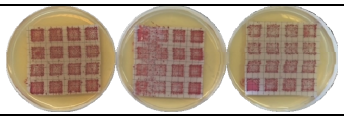
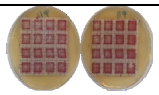
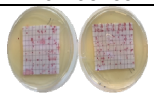
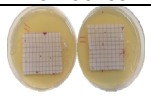
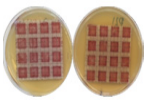
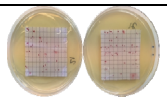
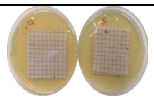
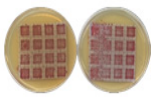
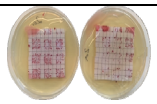
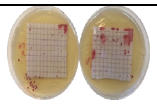
	Agar view	Bacteria amount (CFU)
P1		> 10000 Heavy growth
P2		> 10000 Heavy growth
P3		> 10000 Heavy growth

Table 4. Viability of probiotics on washed samples

	Unwashed	After 3 washes	After 5 washes
P1			
P2			
P3			

In order to investigate the applicability of the fabric, the wettability was explored. To determine the wettability of the fabrics, the water contact angle (Θ) was measured. The effect of the printing parts on the contact angle results of untreated polyester fabric exhibited a high wettability and average contact angle of 42.7° as it is produced with microfibers. Regarding the contact angle results of the printed parts of the treated fabrics, they revealed high angles indicating a low wettability (Table 5).

Table 5. Contact angle results

Sample	Contact Angle θ (°)
Fabric	42.7 ± 4.3
P1	121.8 ± 3.5
P2	118.3 ± 4.4
P3	137.2 ± 2.0

Uddin and Lomas (2010) had the similar results with screen–printed cotton fabrics, their wettability of the fabrics decreased after printing process (19). SEM images of the printed fabrics are shown in Figure 2.

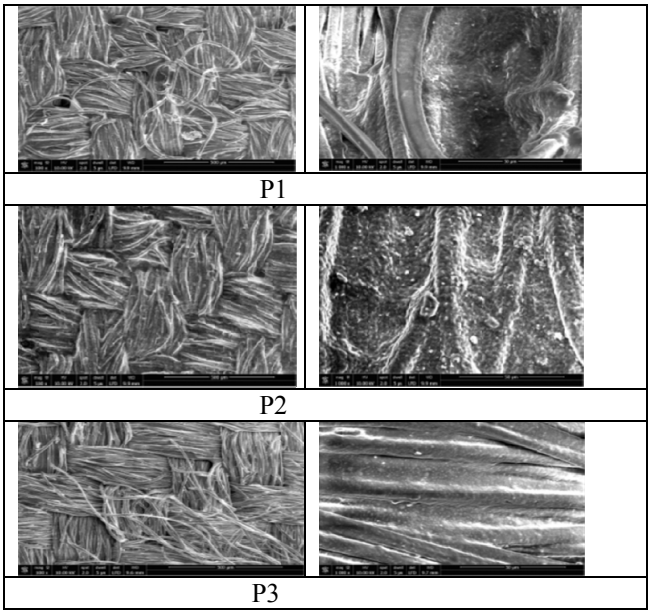


Figure 2. SEM images of the samples

They only give an impression of the quality of the print as none of the printed samples clearly showed a spore like structure. The analysis by SEM revealed that the treated samples showed a homogenous application of the paste. Abrasion resistant of the samples were tested to determine their durability and obtained results showed that the reference fabrics gave the worst results and specimen breakdown occurred after 15000 rubs with broken fibers in warp and weft direction. The printed fabrics didn't reveal broken fibers in both directions after 15000 rubs and only the print intensity decreased.

Table 6. Abrasion resistant of the samples

Rubs	Fabric	P1	P2	P3
2000	5	5	4	5
4000	4	5	4	4
6000	4	5	5	4
8000	4	4	4	4
10000	2	4	4	4
12000	2	3	3	4
14000	2	2	3	3
15000	1	2	2	3

CONCLUSION

Usage of probiotic printed fabrics in the hospital textiles may become efficient tool for preventing HAI which are strictly related to spreading of nosocomial pathogens. In the present study, the aim was to develop strategy to incorporate beneficial spores in fabrics by using screen printing to prevent or reduce the colonization of pathogens on hospital textiles and transmission trough these textiles. The results were shown that all samples exhibited certain amount of viability and about 10^4CFU were counted on each sample. These results showed that the viability of probiotics printed on a textile was achieved by printing a paste containing probiotic agents on a polyester substrate. With the incorporation of beneficial bacteria/spores in the woven fabrics, these fabrics become a reservoir for beneficial

bacteria and may provide an antagonistic/ antimicrobial activity against the contamination of pathogens. Usage of these fabrics in bed linens or uniforms may reduce the infections causing by nosocomial pathogens. Based on data obtained from this study, a further study is underway which will focus on competition tests to determine the inhibition mechanism of printed fabrics with beneficial bacteria/spores more detailed against common pathogens.

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(REFEREED RESEARCH)

IMPROVEMENT OF FLAME RETARDANT CHARACTERISTIC OF RAW SILK FABRIC

HAM İPEK KUMAŞIN GÜÇ TUTUŞURLUK ÖZELLİĞİNİN GELİŞTİRİLMESİ

Zeynep ÖMEROĞULLARI BAŞYİĞİT

Usak University, Faculty of Engineering, Textile Engineering Department, Usak, TR
Corresponding Author: zeynep.omerogullari@usak.edu.tr

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ABSTRACT

In this study, organophosphorus based flame retardant (FR) agent was used in pad-dry-cure-wash treatment in order to enhance the flame retardant characteristics of raw silk fabrics. Silk material is composed of two cores of fibrion surrounded by a layer of sericin which has to be removed from the whole before any chemical finishing treatment. In order to improve the poor wetting capability arising from hydrophobic character of raw silk fabric, degumming process has been widely used for a long time as a necessary process which consume high amount water, chemicals and energy while causing significant weight-loss after the treatment. In today's conditions, water/chemical-saving methods take important part in textile finishing treatments because of environmental concerns. For this aim; low-temperature plasma treatment with various gases at different exposure times in different exposure power were applied on some of the raw silk fabrics before FR finishing process while some of the raw silk fabrics were undergone through conventional degumming processes. Flame retardancy, hydrophilicty, weight losses of the samples were tested, respectively. Morphological analyses of samples were tested by SEM and chemical structures of the fabrics were analyzed by FTIR-ATR. According to LOI test results, it was determined that especially nitrogen plasma pre-treatment had a significant improvement in flame retardancy of raw silk fabric. It was concluded as an output of this study that plasma treatment as a pre-treatment improved the flame retardancy of raw silk fabrics and had a significant effect on increasing wettability properties of raw silk fabrics without the necessity of conventional degumming process.

Keywords: Silk fabric, flame retardant, plasma treatment, wettability

ÖZET

Bu çalışmada, ham ipek kumaşın güç tutuşurluk özelliğini geliştirebilmek amacıyla, organofosfor esaslı güç tutuşurluk maddesi emdirme-kurutma-kondenzasyon-yıkama işleminde kullanılmıştır. Ipek lifinde, fibroin kısmını çevreleyen serisin yapının herhangi bir kimyasal terbiye işlemi öncesinde uzaklaştırılması gerekmektedir. Ham ipek kumaşın hidrofobik özelliğinden kaynaklanan zayıf ıslanabilirlik özelliğini geliştirmek amacıyla, yüksek miktarda su, kimyasal ve enerji harcayan serisin giderme işlemi uzun yıllardır kullanılan gerekli bir işlemdir. Bugünün koşullarında, çevresel kaygılar nedeniyle su-kimyasal tasarruf eden metotlar tekstil terbiye işlemlerinde büyük önem arz etmektedir. Bu amaçla; güç tutuşurluk bitim işlemi öncesinde, farklı güçlerde farklı sürelerde ve farklı gazlarla düşük sıcaklıkta plazma işlemi bazı ham ipek kumaşlara uygulanırken, diğer ham ipek kumaşlara ise konvansiyonel serisin giderme işlemi uygulanmıştır. Numunelerin sırasıyla güç tutuşurluk, hidrofilite ve ağırlık-kaybı testleri yapılmıştır. Morfolojik analizler SEM ile test edilirken, kumaşların kimyasal yapıları FTIR-ATR ile analiz edilmiştir. LOI sonuçlarına göre, özellikle azot plazma ön işleminin ham ipek kumaşın güç tutuşurluk özelliği üzerinde belirgin bir iyileştirme yaptığı belirlenmiştir. Çalışma çıktısı olarak, ön işlem olarak plazma işleminin yapılmasının hem ham ipek kumaşta güç tutuşurluk özelliğinin geliştirdiği ve hem de ayrıca konvansiyonel serisin giderme işlemine gerek kalmadan ham kumaşta ıslanabilirlik özelliğinin artmasını sağladığı sonucuna varılmıştır.

Anahtar Kelimeler: İpek kumaş, güç tutuşur, plazma işlemi, ıslanabilirlik

Corresponding Author: Zeynep Ömeroğullari Başyiğit, zeynep.omerogullari@usak.edu.tr

INTRODUCTION

As it is well known, textiles are the materials that could cause serious fire hazards due to their flammability and ignitibility features. Therefore, there has been an increasing consideration to improve the flame retardant property of textiles for the reduction of fire hazard over the past decades. Silk fabric is widely used as pajamas, evening dress, and domestic furnishing materials for its luster, soft handle, wearing comfort, and aesthetic appearance. However, silk fiber continues to burn and cannot self-extinguish once ignited. Its limited oxygen value (LOI) value is about 23% which still needs flame retardancy finish to fulfill the commercial requirements. It is therefore vital to improve the flame retardant property of silk fabrics in which the safety regulations are concerned. Current safety laws and regulations have raised safety and environmental protection standards on flame retardant (FR) agents and support the development of environmentally friendly structures for replacing the halogen based compounds that cause toxic gases during burning process [1-5]. On the other hand, sericin layer of raw silk fabric has poor functional properties, such as dull appearance, stiff handle and hydrophobic characteristics which prevents penetration of finishing or dyeing chemicals into the fiber. Therefore, degumming process is a necessary treatment before any finishing application of silk materials in order to achieve good functional properties [6-11].The surrounding sericin could be removed by enzymes or in hot aqueous solutions containing soap, alkali and synthetic detergents. However, the methods mentioned above, are thermal- and wet-chemical processes increase the cost in effluents treatment which have environmental concern due to waste water discharge. Furthermore, these methods generally consume high amount of water, chemical and energy. They also have the possibility of degradation or fibrillation of fibroin material and uneven chemical absorption in the finishing bath because of using harsh chemicals at high temperatures in degumming process [12-19]. That's why, in today's conditions; it is important to support the improvement of functional properties of silk fabric with an environmental-friendly method which consumes less energy, water and chemical in finishing treatments.

Low temperature plasma treatments are widely used in surface modifications in polymer and textile industries due their remarkable advantages such as being dry and fast process that uses no water, less energy and less chemical that decreases waste water discharge and providing surface modification without affecting bulk properties of the material which could be named as "effective environmental-friendly method". The excited particles, electrons and photons of plasma have high energies that can initiate various chemical reactions which causes obtaining new functional groups on the surface of the treated material [3,6,20].

The aim of this study was improving flame retardant characteristic of raw silk fabric by increasing the penetration of finishing chemicals into the fibers properly with using an environmental-friendly way instead of conventional degumming process which has to be carried out before finishing treatments. Thus, in this study low-temperature plasma treatment was used with various gases as an alternative method which acted as a pre-treatment before FR finishing of silk fabric while taking the advantage of making surface modification on the raw silk fabric without the necessity of using degumming process which uses harsh chemicals at high temperatures. So, by this way, FR property of raw silk fabric was improved and wettability characteristics of raw silk fabrics were enhanced without using conventional degumming processes.

Experimental Details

Material

100 % silk woven plain fabric which is 39.6 g/m^2 (36 warp/cm x 27 weft/cm**)**, supplied by Ödemiş İpek Company (Turkey) was used in this study. Marseilles soap was taken from İpek Sabun, non-ionic surfactant was supplied by Hunstman Textile Effects (Charlotte, USA), polyphosphate and Na_2CO_3 (99.5 %) were purchased from Sigma Aldrich. These chemicals were used for chemically degumming process to remove sericin from raw silk fabrics. Dialkylphosphonocarboxylic acid amide as a flame retardant agent and Invadine PBN as an anionic surfactant were purchased from Huntsman Textile Effects (Charlotte, USA). Phosphoric acid (85%) was used as a catalyzer and an ionic surfactant added into the finishing bath as a wetting agent.

Method

Degumming Process

Silk fibroin fabric was obtained by removing sericin through a degumming process showed in Fig.1, involving boiling the raw silk fabric at a liquor ratio of 1 : 60 with 4-5 g/L Marseilles soap , 1-1,5 g/L non-ionic surfactant, 1-1,5 g/L polyphosphate and 1g/L Na_2CO_3 at 95 ^{0}C for 45 min at pH 9. After thoroughly washing with warm water, the silk fibroin fabric was dried at room temperature (25 ^{0}C).

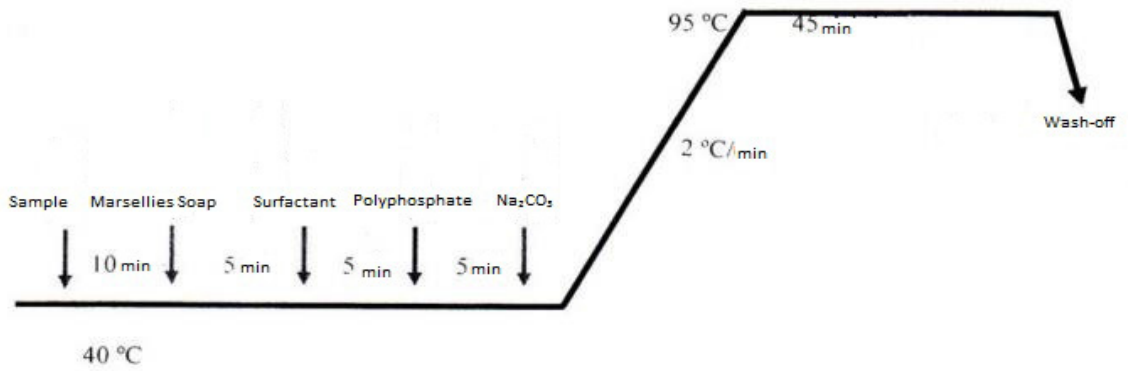

Fig.1. Degumming process of silk fabric

Low Temperature Plasma Treatment

Surface modifications of raw silk fabrics were achieved by low temperature plasma treatment in Diener vacuum plasma (Diener, Germany) in order to investigate the possibility of carrying out plasma treatment for improvement of wetting characteristics of raw silk fabrics instead of degumming silk fabrics with harsh chemicals at hot temperatures before finishing processes. After placing the samples into the plasma device, oxygen, argon and nitrogen gases were discharged into the vacuum chamber, respectively. The process was operated in LF (Low Frequency) generator at a frequency of 40 kHz, at a power of 75 W and 50 W, at a pressure of 0.4 mbar for 1 and 7 min at low temperature (<50^0C). In order to determine the modification effects of plasma treatment and variations in surface characterization of samples, exposure times and power of the treatment were varied in this study. Codes of treated fabrics were showed in Table 1.

Finishing Process

After surface modification and degumming processes, in order to determine the effects of surface modification on the finishing performance of silk fabrics, 250 g/L dialkylphosphonocarboxylic acid amide was used as a flame retardant agent in the recipe of finishing process of treated silk fabrics. 7 g/L phosphoric acid (85%) and 1-1,5 g/L anionic surfactant were used as well in pad-dry-cure system. After applying FR agent in padding bath with a wet-pick-up ratio as 75%, then the fabric was dried at 90^0C for 1-2 min, and then cured at 175^0C for 2 min.In after-wash process, the cured silk fabrics were soaked in water at a liquor ratio of 1:30 containing 2 g/L soap at 60^0C for 20 min, then rinsed with water and then dried at room temperature.

Wicking Hydrophilicty Test

The wicking hydrophilicity test was performed according to DIN 53924. The rising heights of the water of the raw, degummed and plasma-treated raw silk fabric samples with exposure times of 1 and 7 min at a power of 75 W and 50 W, were measured on a scale after 10, 30, 60 and 300 sec.

Weight Loss Test

Weight of treated silk fabrics after treatments were measured on a precision scale and weight losses of the samples calculated as a percentage (%).

Color Spectrum Analysis

Whiteness, yellowness indexes and ΔE values of raw, degummed and plasma-treated raw silk fabrics were calculated by Spectraflash SF 600X Datacolor Reflectance Spectrophotometer in order to determine the changes especially in whiteness and yellowness values of silk fabrics when treated with different gases under specified plasma treatment conditions.

Scanning electron microscope (SEM)analysis

The surface morphology of untreated and treated silk fabrics were scanned by a Hitachi S-3200N electron microscope under a high vacuum at 1500 magnification after being coated with gold-palladium (Au-Pl) at a thickness of 40–50 nm.

Limited Oxygen Index (LOI) Test

The limited oxygen index (LOI) test of samples was performed according to the standard ASTM D 2863-77 after FR finishing process. The numerical index, LOI, is defined as the minimum concentration of oxygen in an oxygen–nitrogen mixture required to just support the downward burning of a vertically mounted test specimen. Thus, a larger LOI value represents a better flame retardancy.

Fourier transform infrared (attenuated total reflectance) spectra FTIR-ATR analysis

FT-IR ATR spectra of the untreated and FR treated silk fabrics were investigated by a Nicolet 510P device in a wave number range of 525–4000 cm^{-1}.

Table1. Codes of treated samples

OP1	Oxygen Plasma 50 W 1 min	**NP1**	Nitrogen Plasma 50 W 1 min	**AP1**	Argon Plasma 50 W 1 min
OP2	Oxygen Plasma 50 W 7 min	**NP2**	Nitrogen Plasma 50 W 7 min	**AP2**	Argon Plasma 50 W 7 min
OP3	Oxygen Plasma 75 W 1 min	**NP3**	Nitrogen Plasma 75 W 1 min	**AP3**	Argon Plasma 75 W 1 min
OP4	Oxygen Plasma 75 W 7 min	**NP4**	Nitrogen Plasma 75 W 7 min	**AP4**	Argon Plasma 75 W7 min
FR-OP1	FR-treated raw silk fabric after oxygen plasma at 50 W for 1 min	**FR-NP1**	FR-treated raw silk fabric after nitrogen plasma at 50 W for 1 min	**FR-AP1**	FR-treated raw silk fabric after argon plasma at 50 W for 1 min
FR-OP2	FR-treated raw silk fabric after oxygen plasma at 50 W for 7 min	**FR-NP2**	FR-treated raw silk fabric after nitrogen plasma at 50 W for 7 min	**FR-AP2**	FR-treated raw silk fabric after argon plasma at 50 W for 7 min
FR-OP3	FR-treated raw silk fabric after oxygen plasma at 75 W for 1 min	**FR-NP3**	FR-treated raw silk fabric after nitrogen plasma at 75 W for 1 min	**FR-AP3**	FR-treated raw silk fabric after argon plasma at 75 W for 1 min
FR-OP4	FR-treated raw silk fabric after oxygen plasma at 75 W for 5 min	**FR-NP4**	FR-treated raw silk fabric after nitrogen plasma at 75 W for 7 min	**FR-AP4**	FR-treated raw silk fabric after argon plasma at 75 W for 7 min

RESULTS AND DISCUSSION

Wicking Hydrophilicity Test Result

Rising height of water after 10,30,60 and 300 seconds of raw and treated silk fabrics were showed in Fig. 2,3,4. As seen below, while raw silk fabric showed a hydrophobic character, plasma-treated raw silk fabrics with different gases enhanced the hydrophilic character of the raw silk fabrics. As seen in Fig. 2, it was determined that, argon-plasma treated silk fabrics showed the minimum increase even at highest exposure time among other plasma treated silk fabrics. In oxygen-plasma treatment showed in Fig.3, increasing both exposure time and power provided more hydrophilic property of raw silk fabrics. When it was analyzed, it could be clearly seen that nitrogen-plasma seen in Fig. 4, had the most significant effect on enhancing wettability properties of raw silk fabrics which the exposure time had more dominant effect compared to effect of plasma power. After 300 sec, the height of water of degummed silk fabric is 10.55 cm that is nearly the same as NP4 (nitrogen-plasma treated raw silk fabric at 75 W for 7 min) which is considered as low temperature plasma treatment could be a better alternative for improving wettability characteristics of raw silk with the advantages of being dry and environmental-friendly process due to consuming less water, chemical and energy, instead of carrying out degumming process with harsh chemicals at high-temperatures. The increases in hydrophilic property of raw silk fabrics attributed to new functional groups such as hydroxyl, carboxyl groups obtained on the surface of silk fabrics via plasma treatment [21-23].

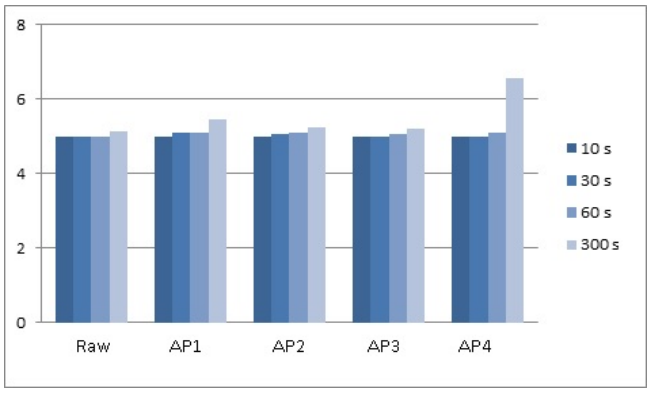

Fig.2. Wicking hydrophilicity results of raw and argon-plasma treated raw fabric

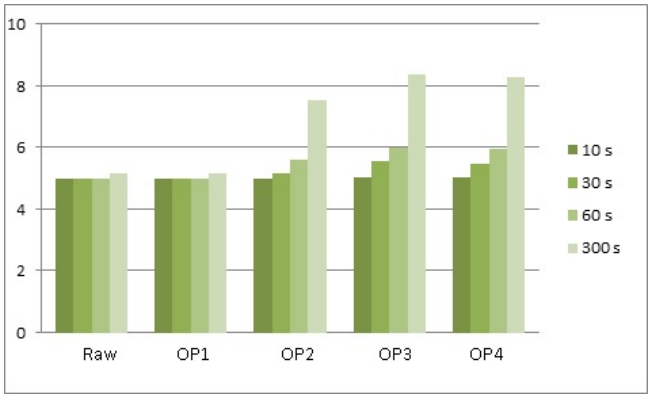

Fig.3. Wicking hydrophilicity results of raw and oxygen-plasma treated raw fabrics

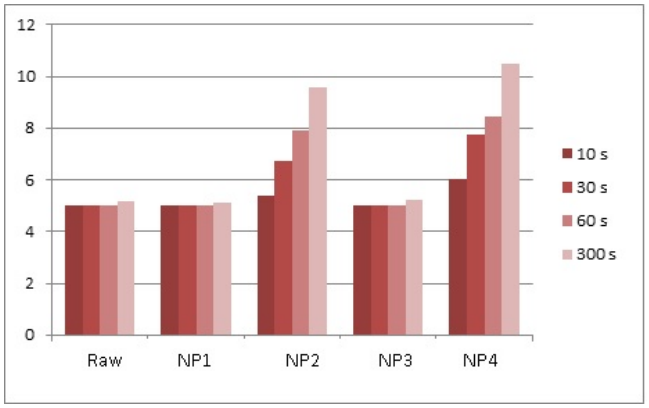

Fig.4. Wicking hydrophilicity results of raw and nitrogen-plasma treated raw fabrics

Color Spectrum Analysis Result

According to the color spectrum analysis; ΔE, yellowness and whiteness indexes of raw, degummed and plasma-treated raw silk fabrics were showed in Table 2. When ΔE of samples were investigated, it was clearly seen that degummed silk fabric had the highest ΔE value among other samples which were treated via plasma process. It was considered that this result was observed because of harsh chemicals usually used in degumming process at high temperatures around 95 ^{0}C, especially for removing sericin layer on it. According to ΔE values, plasma-treated silk high temperature of process around fabrics had less color changes on raw silk fabrics compared to degumming process which was attributed to the mechanism of the plasma process that did not use either chemical or high temperature and had no alterations on bulk properties of treated material but just had a modification effect on surface of raw silk fabrics. However, these ΔE values of plasma-treated fabrics were at certain extent, which was attributed to the effect of enhanced surface roughness by plasma treatment that causes a decrease in amount of reflected fraction of lights from the treated rough surfaces of silk fabrics when compared with untreated smooth surfaces. When whiteness and yellowness indexes were analyzed, it could be indicated that generally plasma-treatment increased the yellowness indexes and decreased the whiteness indexes of silk fabrics which could be considered as it did not have a favorable effect on whitening raw silk fabrics. This increase in yellowness was more obvious when the exposure time and power of nitrogen-plasma were increased. This result was attributed to the formation of chromophoric products containing carbonyl groups on the surface of the material [24].

Weight Loss Test

Weight of raw, degummed and plasma-treated raw silk fabrics and calculated weight losses as percentages (%) were showed In Table 3. As it is seen clearly in the Table 3 that the highest weight loss was belong to degummed silk fabric with the value of 9.30 %. When plasma-treated samples were examined, the highest weight loss (with 6.77 %) of samples among plasma-treated fabrics, was showed in nitrogen-plasma treated raw silk fabrics which were in agreement with Fig. 4 of wettability results of nitrogen plasma-treated raw silk fabrics. It was considered that

plasma etching effect on the surface of the material caused removing sericin from the silk fabric resulting in weight loss of raw silk fabrics. However, it should be emphasized that while both treatment (degumming and nitrogen-plasma treatment) achieved nearly the same level of wettability on raw silk fabrics with the value of 10.55 cm, plasma treatment caused less weight-loss compared to degumming process.

Table 2. ΔE, yellowness and whiteness indexes of raw, degummed and plasma-treated raw silk fabrics

Samples	Yellowness Index (ASTMD1925)	Whiteness Index (Stensby)	ΔE
Raw	18.964	59.606	-
Degummed	12.591	69.284	3.055
OP1	18.328	58.328	1.572
AP1	19.286	58.812	1.714
NP1	19.274	58.664	1.377
OP2	19.047	58.529	1.478
AP2	18.869	58.819	1.317
NP2	23.707	52.957	2.690
OP3	19.742	57.991	1.545
AP3	18.268	58.536	1.100
NP3	20.836	56.418	1.805
OP4	20.378	57.641	1.396
AP4	21.514	55.531	1.908
NP4	23.009	53.524	2.271

LOI Test Results (%)

Limited oxygen index values (%) of raw, degummed and FR treated after plasma treatment of raw silk fabrics were showed in Table 4. As seen in Table 4, raw or degummed silk fabric had no flame retardant effect before finishing process. After padding degummed silk fabric with phosphorus based FR agent, LOI value was increased up to 29.6 %. When FR-treated silk fabrics which were pre-treated with different plasma gases were examined, it could be clearly seen that all plasma applications which were used as pre-treatment before FR finishing, provided enhanced FR characteristics of raw silk fabrics. These results were attributed to the positive effect of plasma treatments on improving wettability properties of silk fabrics due to new functional groups obtained on the surface of the material, so that it increased penetration of FR agent into the silk fabric. It was indicated that highest LOI values were belong to nitrogen-plasma treated raw silk fabric. It was considered that this result could be attributed to the positive synergetic effect between nitrogen-phosphorus compounds on the treated silk fabric.

Results of SEM analysis

SEM micrographs of raw, degummed, plasma-treated and FR-treated after nitrogen plasma treatment at 75 W for 7 min were showed in Fig. 5, respectively. According to the SEM results, it could be clearly seen that untreated raw silk fabric (Fig. 5.a) had a smooth surface whereas fibrillation and degradation was occurred on degummed silk fabric (Fig. 5.b) due to removing sericin layer which keeps the fibroin macromolecules together. During conventional degumming method of raw silk fabric, surrounding sericin is hydrolyzed and the amide bonds of the long protein molecules are broken into smaller fractions, then dissolved in degumming bath containing alkali, soap or synthetic detergents. However, the higher temperature (95 °C) and an alkaline pH (8–9) in the presence of harsh chemicals in the treatment process impose a remarkable unnatural environment on the silk which causes partial degradation of fibroin [8,12,19]. So higher weight losses of silk fabrics after degumming process could be attributed to the harsh parameters taking part in traditional degumming processes. Some chemical residues of FR finishing and also partial roughness of surface modification obtained by plasma treatment could be seen In Fig. 5.d.When nitrogen (Fig.5.c), argon (Fig.5.e) or oxygen plasma treatments (Fig. 5.f) were carried out, roughness could be clearly seen on the surfaces of raw silk fabrics. This result was attributed to the etching effect caused by the physical bombardment of the energetic plasma species on the substrate surface such as fluting and/or grooving observed on materials [3,6].

Table 3. Weight loss (%) of raw, degummed and plasma-treated raw silk fabrics

Samples	Weight Loss (%)	Samples	Weight Loss (%)	Samples	Weight Loss (%)
OP1	5.08	**NP1**	5.93	**AP1**	4.23
OP2	5.08	**NP2**	6.77	**AP2**	4.23
OP3	5.50	**NP3**	5.93	**AP3**	3.38
OP4	5.93	**NP4**	6.77	**AP4**	5.93
Degummed	9.30				

Table 4.LOI results (%) of raw, degummed and FR treated after plasma treatment of raw silk fabrics

Raw	23.8		**Degummed**	23.2	
			Degummed-FR	29.6	
AP1-FR	26.2	**OP1-FR**	26.2	**NP1-FR**	27.6
AP2-FR	26.2	**OP2-FR**	28.5	**NP2-FR**	30.4
AP3-FR	26.4	**OP3-FR**	29.6	**NP3-FR**	32.3
AP4-FR	28.5	**OP4-FR**	31.1	**NP4-FR**	33.1

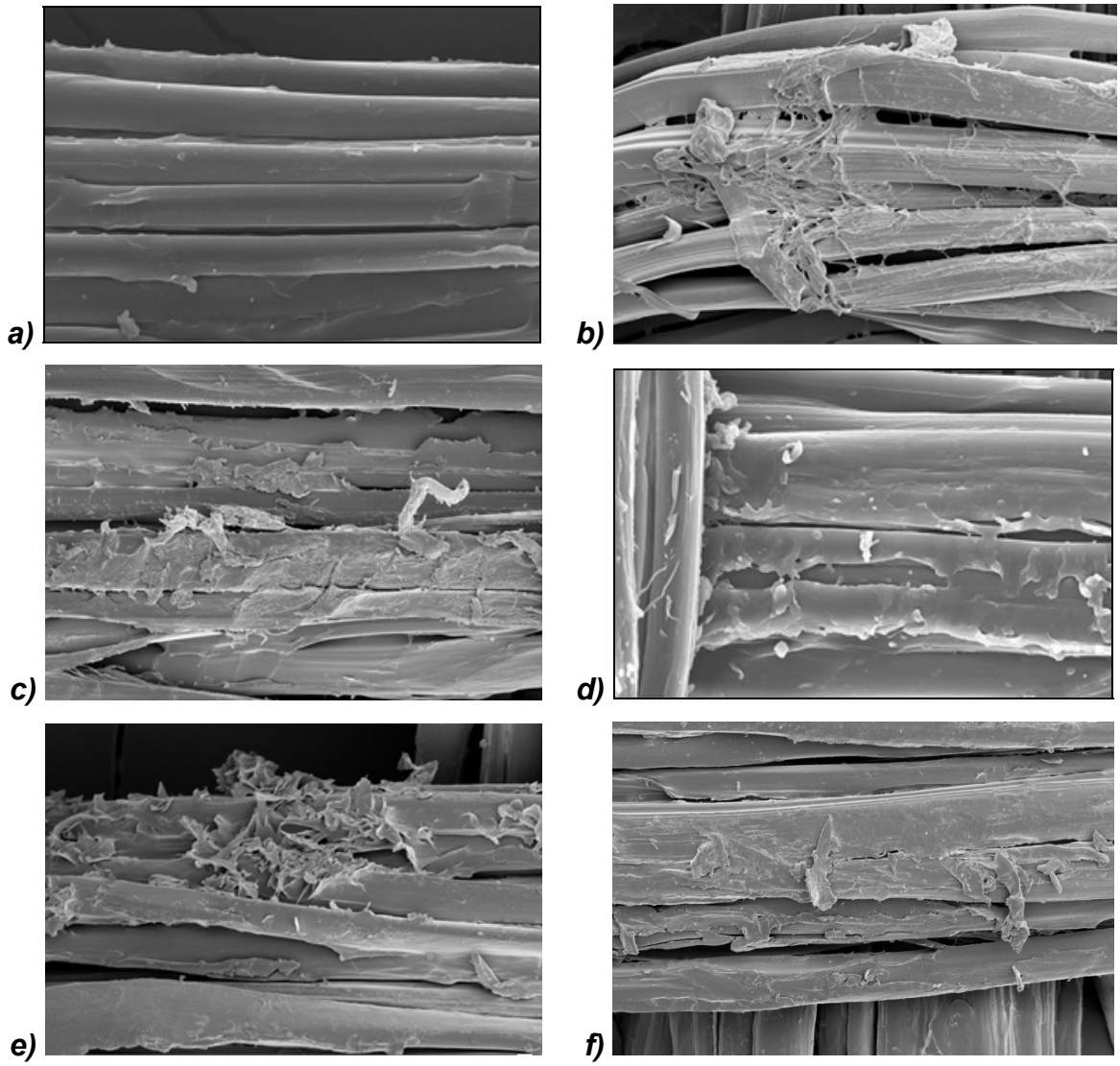

Fig 5. SEM micrographs of raw, degummed and plasma-treated raw silk fabrics a)Raw silk fabric b)Degummed silk fabric c)NP4(Nitrogen-plasma treated raw silk fabric at 75 W for 7 min) d) FR-NP4 (FR-treated after nitrogen-plasma treatment) e) AP4(Argon-plasma treated raw silk fabric at 75 W for 7 min) f)OP4 (Oxygen-plasma treated raw silk fabric at 75 W for 7 min)

Fourier transform infrared (attenuated total reflectance)(FTIR-ATR) analysis results

FTIR-ATR analysis of raw silk fabric, nitrogen-plasma treated raw silk fabric at 75 W for 7 min (NP4) and FR-treated silk fabric after nitrogen plasma application (FR-NP4) were showed in Fig. 6. The FTIR-ATR spectra showed characteristics bands at 1622 cm^{-1} (amide I), which is due to the β confirmation of the crystalline region, while the band appearing at 1515 cm^{-1} (amide II) is due to the random coil conformation of fibroin molecules. The peaks that appeared at 1440 cm^{-1} are attributed to the presence of an amino acid group of CH-stretching of CH_3 deformation of the silk. The appearance of the absorption bands at 3288 cm^{-1} is caused by the free-OH stretching and NH- stretching vibrations [25].

Although for plasma-treated raw silk fabric, maximum effective plasma gas at highest plasma parameters were chosen according to the other performance tests in this study, no significant difference were observed between FTIR-ATR spectra of untreated raw silk fabric and plasma-treated raw silk fabric. This result was attributed to the mechanism of the plasma application which process modification only on the surface of the material without effecting bulk properties. It was also indicated in the literature [26] that infrared equipment by itself may not be the best one to identify chemical changing on the low frequency plasma treated sample's surface unless wet chemical process was carried out after surface modification. However, for the sample of FR-NP4 which had the FR treatment after nitrogen-plasma application, strong band could be seen around 1057 cm^{-1} which indicates P–O–C and C–O–C stretching vibration. Another peak around 1227 cm^{-1} also indicates the absorption overlap of P=O and amide III of silk fibres [4] which caused by phosphorus based FR agent used in finishing treatment.

CONCLUSION

In this study, in order to improve the FR property of silk fabrics; phosphorus based FR agent was used in finishing bath after nitrogen, argon and oxygen-plasma application

which is carried out as pre-treatment. On behalf of making a comparison between conventional method and plasma treatment, some of the samples were undergone through conventional degumming process. After the plasma and degumming treatments, wetting effect of the processes, color spectrums of treated samples and weight losses were investigated. According to the results, it was observed that nitrogen-plasma application was more effective than the other plasma gases on improving the wettability effect of raw silk fabric. When weight losses were examined, it was indicated that degumming processes caused more weight-loss than plasma treatments carried out with various gases. After FR finishing processes, LOI values were examined and it was determined that especially nitrogen-plasma pre-treatment supported the improvement of FR characteristic of raw silk fabrics. In the study, it was considered that plasma treatment could be a better alternative for being used as a pre-treatment of raw silk fabrics when compared to conventional processes.

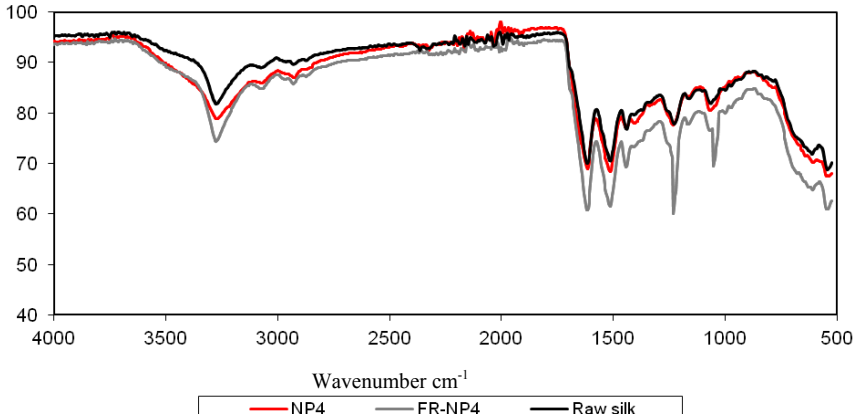

Fig.6.FTIR-ATR analysis of raw silk fabric, nitrogen-plasma treated (NP4), FR-treated silk fabric after nitrogen-plasma application (FR-NP4)

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(REFEREED RESEARCH)

CHEMICAL TREATMENT OF CHICKEN FEATHER PRIOR TO USE AS FILLING MATERIAL

DOLGU MALZEMESİ OLARAK KULLANIM ÖNCESİ TAVUK TÜYLERİNİN KİMYASAL MUAMELESİ

Umut Kivanc SAHIN

Istanbul Technical University, Faculty of Textile Technologies and Design, Istanbul, 34437 Turkey

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ABSTRACT

In this study, an alternative combined method to treat chicken feathers to be used as a filling material has been developed. The proposed method for treatment of chicken feathers is based on the combination of aldehyde treatment and acid-alum method including less harmful chemical usage with fewer number of processing steps. A number of international standard tests such as oxygen index number, turbidity, moisture, content analysis (composition), odor test have been applied on the treated chicken feathers in order to determine whether it is suitable to be used as a filling material. Results showed that with less harmful chemical usage, presenting chicken feathers with fluffy structure and almost white in color after treatment, the proposed method offers a good alternative in treatment of chicken feathers by providing the demanded physical properties.

Keywords: Chemical treatment, chicken feather, filling material, oxygen index number, turbidity

ÖZET

Bu çalışmada, dolgu malzemesi olarak kullanılacak olan tavuk tüylerinin terbiyesi için alternatif kombine metot geliştirilmiştir. Tavuk tüylerinin terbiyesi için önerilen metot, aldehit ve asit-şap metotlarının kombinasyonuna dayalı olup daha az sayıda işlem adımı ve daha az zararlı kimyasalların kullanımını içermektedir. Dolgu malzemesi olarak kullanım uygunluğunun tespit edilmesi için terbiye işlemi gören tavuk tüylerine, oksijen indeks sayısı, bulanıklık, nem, içerik analizi (tüy kompozisyonu) ve koku testi gibi birçok uluslararası standart test uygulanmıştır. Sonuçlar, önerilen metodun az zararlı kimyasal kullanımı ile birlikte kabarık yapıda ve neredeyse tamamen beyaz renkte tavuk tüyü elde edilmesini sağladığını ve istenen fiziksel özellikleri sağlayarak tavuk tüyünün terbiyesinde iyi bir alternatif oluşturduğunu göstermektedir.

Anahtar Kelimeler: Kimyasal muamele, tavuk tüyü, dolgu malzemesi, oksijen indeks sayısı, bulanıklık

Corresponding Author: Umut Kıvanç Şahin, sahinumut3@itu.edu.tr

1. INTRODUCTİON

Filling materials are generally used for trapping air and providing insulation. The insulation and comfort levels mainly depend on the type and amount of filling material as well as on filling method.

There are two types of filling materials: i) synthetic fibers and, ii) natural fibers. Polyester is the most common synthetic filling fiber because of its non-allergic and anti-odor properties. Moreover, it has low thermal conductivity, good compressibility, and low moisture absorption. The insulation value depends on polymer's thickness, density, uniformity and bonding techniques. The transformation parameters from polymers to staple fiber like crimp, texture, cross-sectional shape, size and number also affect the insulation properties. Apart from synthetics, duck and goose feathers are the most common natural filling fibers, but they are highly expensive compared to synthetics. Down filling materials are the mixture of down components including nestling down, plumules, down fibers, feathers and feather residue. Down is light, thin, soft, and fluffy [1, 2]. Down, nestling down, and plumules are the most valuable parts of any plumage product. Their structure is three dimensional and since their clusters are composed of interlocking fine barbs, they attach to each other and thus create millions of tiny air spaces trapping air. Down, nestling down, and

plumules are defined as down cluster which is a new term proposed by the IDFL. On the other hand, the detached barbs from down or the basal end of waterfowl feather quill shafts are down fibers whereas the outgrowth forming the contour and external covering of fowl is defined as the feather [2]. Having two dimensional structures, down fibers and feathers cannot provide efficient insulation as down clusters. However, down cluster entangles when wet and causes decrease in insulation quality and drying behavior. Moreover, after a while, unpleasant odor and allergic reactions might occur with the entangled down clusters.

Apart from duck and goose feathers, chicken feathers can be alternative natural option due to lower price and high availability as being waste of a highly consumable product in food industry [3]. Moreover, duck and goose feathers are collected from the animals when they are alive, and there is growing public opposition against use of such feathers.

Chicken feathers have round hollow quill from which the fine, thread-like barbs extend on both sides, branching to form barbules. Through the hooklets on the barbules, the barbs and barbules interlock to form a network containing a large number of air channels. Thanks to this structure, when used as filling material in beds, it causes an insulating air layer between the body and the surrounding which results in warmth in cold weather. Fine air channels together with hydrophilic feathers further contribute to better transport of moisture [4]. However, the fundamental difference compared to goose and duck feathers is their thinner and straighter quills which cause lower elasticity, less insulating capacity and sensitivity to stress [5].

Chicken feathers have wide usage in technical textiles applications, and keratin in chicken feather has been used for manufacturing artificial hair. The behavior of the chicken feather is mainly studied by the researchers as a reinforcing material for composite structures [6-12]. In order to increase the elasticity, insulation and strength, chicken feathers are chemically treated. There are two very popular approaches, namely aldehyde treatment based on a patented process [13] and acid-alum method based on a Keracurl ®treatment [14]. Aldehyde treatment method includes removal of impurities by washing, treatment with a haemolytic agent and a second washing using nonyl-phenol non-inonic detergant followed by keratin and aldehyde treatment to which chromium fluoride is added as well. [13]. The treatment is finalized with a number of rinsing cycles. Haemolytic agents cause increase in rate of red blood cell destruction, and its mechanism is explained in detail elsewhere [15]. High rate of red blood cell destruction may cause haemolytic anaemia [16]. Nonyl-phenol detergents are banned in many countries, and chromium fluoride is a known toxic chemical. It is apparent that aldehyde treatment of chicken feathers comprise use of many hazardous chemicals, and high level of protection as well as expertise is demanded during chicken feather treatment using aldehyde treatment method. On the other hand, acid-alum method comprises plasticization of chicken feathers in diluted acid and alum solution. Sulfuric acid is used, and the treatment time is at least 90 minutes, followed by a 20 minutes washing step at 60-70^{0}C with soda ash, and several rinsing steps until chicken feathers are completely neutralized and free from residual chemicals [14]. In general, washing process is conducted with running water and partial C_2H_5OH to obtain clean, sanitized and odor free chicken feathers [3]. Acid-alum method is a timely process, and high amount of waste water must be treated prior to discharge. Moreover, sulfuric acid is corrosive, and demands special equipment during use and storage. Both methods include use of expensive and environmentally harmful chemicals and lots of water, thus an alternative comprising fewer treatment steps and use of less harmful chemicals is highly demanded. In this study, an alternative combined method to prepare chicken feathers to be used as a filling material has been developed.

2. EXPERİMENTAL

2.1. Materials

Chicken Feathers are kindly supplied as biowaste from a chicken food company. The chemicals used for processing and testing include EDTA (chelating agent, Mw= 372.240 g/mol), aluminum sulfate-18- hydrate (Mw= 666.420 g/mol), sodium carbonate (Mw= 105.989 g/mol), acetic acid (Mw= 60.05 g/mol), potassium permanganate (Mw= 158.034 g/mol), trichloroethylene (Mw=131.401 g/mol) supplied by Merck® and ECE standard reference detergent including linear sodium alkyl benzene sulphonate (8%), ethoxylated tallow alcohol (2.9%), sodium soap (3.5 %), sodium tripolyphosphate (43.7%), sodium silicate (7.5%), magnesium silicate(1.9%), carboxy methyl cellulose(1.2%), etylene diamine tetra acetic acid (0.2%), sodium sulphate (21.2%) and water (9.9%). All chemicals used are reagent grade with the exception of standard reference detergent. All concentrations are expressed as % (weight /weight). Plain weave cotton fabric with a basis weight of 140 g/m^2 (32 ends/cm x 36 ends/cm) was used to produce cotton fabric bags for loading chicken feather inside the washing machine.

2.2. Chemical Treatment Method

The proposed method for preparation of chicken feathers used in this study is based on the combination of aldehyde treatment and acid-alum methods. Aldehyde treatment comprises soaking of chicken feathers for 10 minutes at 40-50^{0}C in 1 g/L of haemolytic agent solution, laundering with 0.25% nonyl-phenol non-ionic detergent at 40-50^{0}C for 20 minutes (or dry cleaning), treatment with 1% $Na_3PO_4.12H_2O$ at 40-50^{0}C up to 30 minutes at pH 6-7 with keratin as stabilizing agent, immersion in 1% alpha-hydroxyadipaldehyde + 0.1% $Al_2(SO_4)_3$ + 0. 05% chromium fluoride (pH 2-4) at 40-50^{0}C for 30 minutes, followed by rinsing and drying in open air. On the other hand, acid-alum method comprises plasticization of chicken feathers in diluted acid and alum solution. The acidic treatment solution is prepared by addition of water, followed by stepwise addition of 2% sulfuric acid and 2% aluminum sulfate (or alum). The chicken feathers are digested in the acidic solution for at least 90 minutes, and then the solution is discharged. The feathers are then treated for at least 20 minutes at pH 5-6 with mechanical agitation in water at 60-70^{0}C, and soda ash is added in order to precipitate aluminum hydroxide. Following warm water treatment, the feathers are rinsed several times until the precipitate disappears. Finally, the feathers are rinsed thoroughly until completely free from acid, and dried.

In order to offer an acceptable alternative process, some steps from both methods were selected. Moreover, alternative chemicals were used in place of harmful ones. EDTA was used in place of haemolytic agent, and ECE standard detergent was used in place of nonyl-phenol non-ionic detergent. The selected steps from both methods as well as alternative chemicals selected are summarized in Figure 1. The developed method alternative to aldehyde and acid-alum methods is given in Figure 2. According to the developed method, firstly, 4 Kg of feather was put in 25 L containers with 20 L water and 3 g/L of standard detergent, 3g/L of chelating agent were added stepwise, and feathers were kept in the container for 1 hour without mechanical agitation. The chicken feathers are filtered and the wash liquor is collected for reuse in laundry. Cotton fabric bags of selected sizes are stitched, and chicken feathers were loaded in the bags after filtering. The bags filled with feather were laundered at 60°C at 600 rpm for 1 hour by using the collected wash liquor. Aluminum sulfate solution at pH 6 was prepared using acetic acid. After laundry, the feathers are kept in 2% aluminum sulfate solution for 5 minutes, and further kept for 5 minutes after addition of 1% sodium carbonate into the solution. After filtering and rinsing, feathers are treated with trichloroethylene at room temperature to remove oily stains. Finally, the feathers are filtered and left to dry at room temperature for 24 hours.

2.3. Analysis Methods

In order to present that the alternative chicken feather treatment method offers treated chicken feathers that are acceptable to be used as filling material, a number of tests were performed, and their results were presented.

2.3.1. Oxygen Index Number Test

The oxygen index number indicates product cleanliness by providing a measurement of oxidizable and soluble material present in the filtrate of the aqueous extract of the material. Chicken feather plumage samples were put into water and tumbled at room temperature for 60 min. The resulting suspension was filtered and mixed with sulphuric acid. Then, the excess water from the chicken feathers was titrated with potassium permanganate. The test was done according to BS EN 1162:1999 test standard [17].

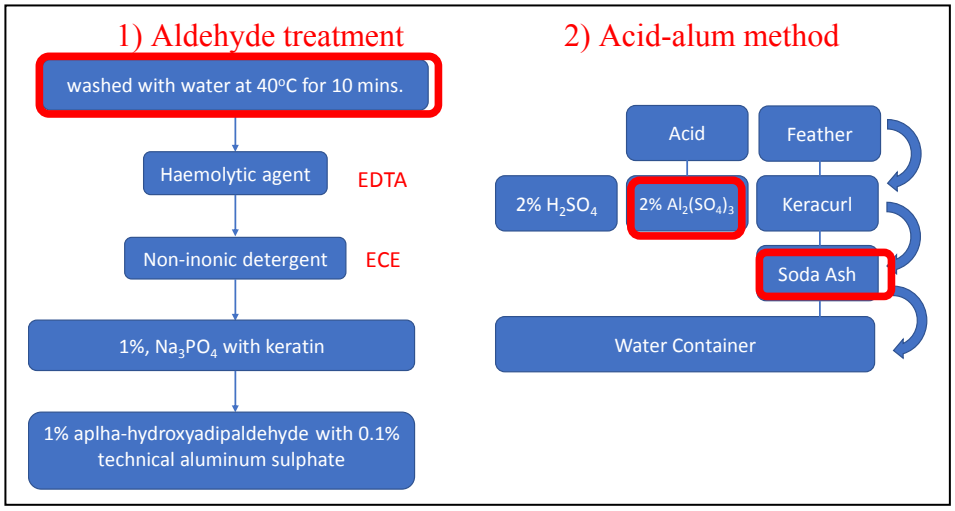

Figure 1. Aldehyde treatment, acid-alum method for preparation of chicken feather together with selected steps and alternative chemicals

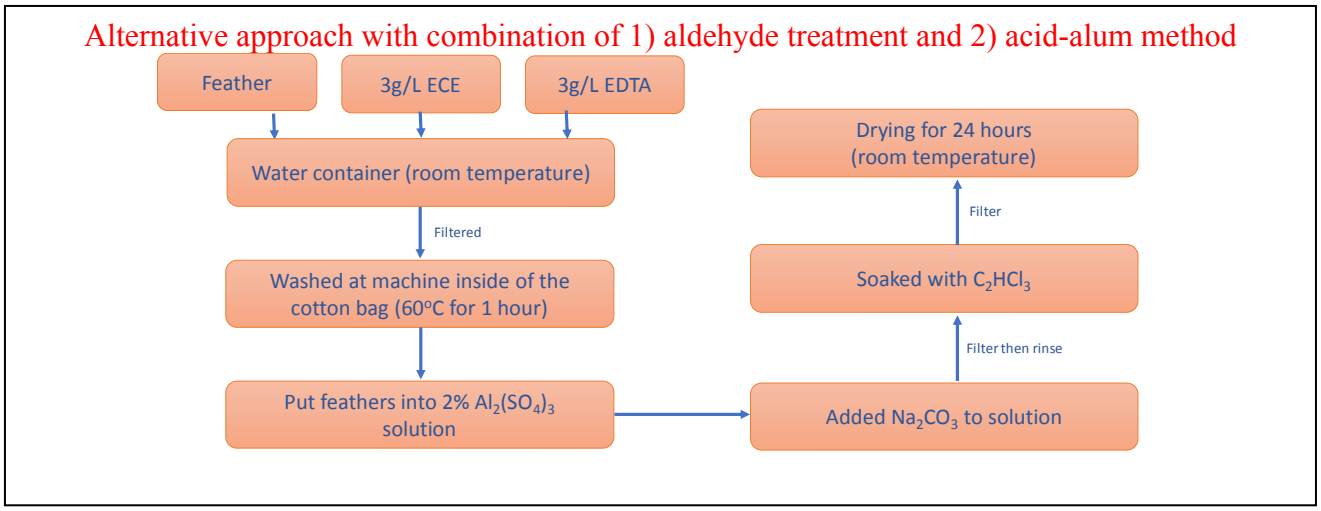

Figure 2. The developed method for treatment of chicken feather

The oxygen index number is calculated as follows:

$$I = 80 \times (A-B) \quad (1)$$

where:

I is the oxygen index number; A is the quantity of 0,02 mol/L potassium permanganate used for the test specimen, in milliliters; B is the quantity of 0,02 mol/L potassium permanganate used for the blank test, in milliliters.

2.3.2. Turbidity

Turbidity shows the reduction of transparency of the filtrate suspension caused by the presence of undissolved and dissolved matter. 10 g of conditioned test specimens were put into a tumble jar containing 1 L of water, and tumbled for 60 minutes followed by filtration. The filtrate is poured into container in order to observe the corresponding height of the poured filtrate in the container when the cross is invisible. This procedure was done according to BS EN 1164:1999 test standard [18].

2.3.3. Moisture Content

Moisture content is the percentage of water held in a sample. 5 grams of the plumage is transferred to uncovered container for testing. The container is then placed and covered in the oven and dried for 130 minutes. After cooling to room temperature the covered container is weighed. The procedure has been repeated until a constant mass, within 1 mg, is obtained. The moisture content is determined from the weight of the dried sample. This procedure was done according to BS EN 1161:1996 test standard [19].

The moisture content expressed as a percentage is calculated as follows:

$$M_c\ \% = [(A-B) / (A-C)] \times 100 \quad (2)$$

where;

M_c is the moisture content in percent; A is the mass of the container with lid and undried individual sample; B is the mass of the container with lid and dried individual sample; C is the mass of the container with a dried lid.

2.3.4. Content Analysis

A mixture of random samples from the chicken feathers was placed in the sorting cabinet and was separated by hand into the following components: i) Down clusters, plumules, ii) Down fiber, iii) Feather fiber. Each component was weighed to the nearest 0.2 mg, and in the case of the double tests, the results were averaged. This procedure was done according to BS EN 12131:1998 test standard [20].

2.3.5. Odor Test

The odor test indicates potential odor problems in a filling material. Material was soaked in water and warmed for 24 hours according to IDFL (International Down and Feather Laboratories) Testing Regulations. Then, material was smelled to determine if a "putrid" condition exists. 15 female and 15 male non-smokers of ages ranging between 18 and 25 participated in odor test. The participants were asked to grade the smell as “putrid smell”, “very bad smell”, “bad smell”, “faint smell”, or “no putrid smell”.

3. RESULTS

A big visual difference is observed between the chicken feather samples before and after application of the proposed chemical treatment. Before chemical treatment, the feathers aggregated together, looked yellowish accompanied with a bed smell. However, after chemical treatment, they became fluffy and almost white in color. Moreover, no disturbing smell was observed. The differences between chicken feather samples before and after chemical treatment can apparently be seen in Figure 3.

The oxygen index number test results are shown in Table 1. According to test results based on IDFL regulations and EN 1162, the oxygen index number values were within the acceptable range for chicken feathers to be used as filling materials.

Figure 3. Images of chicken feather before and after chemical treatment

Table 1. Oxygen index number test results

Samples #	Potassium Permanganate used (mL)	Oxygen Index Number
Sample 1 (distilled water)	0.22	13.6
Sample 1 (chicken)	0.39	
Sample 2 (distilled water)	0.27	12.0
Sample 2 (chicken)	0.42	
Sample 3 (distilled water)	0.25	12.8
Sample 3 (chicken)	0.41	

Turbidity test results showed that although all the filtrate was poured the cross was visible throughout the test, which means that no turbidity occurred. Test results indicated that the chicken feather was completely free from impurities after treatment according to newly developed method.

The moisture content values of chemically treated chicken feathers are given in Table 2. According to test results based on IDFL regulations and EN 1162, the moisture content values were within the acceptable range for chicken feathers to be used as filling materials.

Table 2. Moisture Content Test Results

Moisture Content (%)	A	B	C
4.23	13.36	12.07	7.84

According to content analysis test results, it was found that the chicken feather consisted of approximately 30% down clusters and plumules, %30 feather fiber, and %40 down fiber. The content analysis showed that there was sufficient distribution among the three types such that insulation would be achieved without entanglement of the feather even when wet.

Prior to treatment, all participants of the odor test reported that the material had “putrid smell” or “very bad smell”. However, the responses just after the treatment and 24 hours after the treatment were all “no putrid smell”. Odor test results showed that no putrid smell was observed after 24 hours which indicates that such treated chicken feather can be suitable for outdoor applications.

CONCLUSIONS

Although duck and goose feathers are commonly used filling materials with superior insulation and comfort properties, there is growing public opposition against their use mainly due to their harvesting of feathers being carried when animals are alive. Synthetic fibers offer a weak nominee as their waste management is an environmental issue. Considering high amount of chicken feather bio-waste coming from food industry, chicken feathers may offer a good natural alternative if they are treated properly. In this study, a chemical process is developed for treatment of chicken feathers with demanded physical properties to be used as filling material. The treated chicken feathers were shown to achieve no turbidity, demanded moisture, acceptable oxygen index number with no disturbing odor, almost white color and fluffy structure. Moreover, offering less harmful chemicals and fewer processing steps when compared to conventional chicken treatment methods, it can be used as alternative method for treatment of chicken feathers to be used as filling material.

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(REFEREED RESEARCH)

SKIN-FABRIC FRICTION AND OTHER PERFORMANCE CHARACTERISTICS OF SOCKS FABRICS PRODUCED FROM CELLULOSIC FIBERS

SELÜLOZİK LİFLERDEN ÜRETİLEN ÇORAPLARIN DERİ-KUMAŞ SÜRTÜNMESİ VE DİĞER PERFORMANS ÖZELLİKLERİ

Sibel KAPLAN, Ceren KARAMAN

Suleyman Demirel University, Textile Engineering Department, Isparta, Turkey.

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ABSTRACT

Skin friction and some performance characteristics of single jersey fabrics produced from cotton (combed, carded and rotor) and regenerated cellulosic fiber (viscose, bamboo, modal, micromodal, Tencel®, Tencel LF® and some blends) yarns were investigated by surface friction, bursting strength, abrasion and pilling resistance tests. Daily or functional socks were the intended end use of the selected materials. According to the results, cotton, 48/52% cotton/modal and Tencel® fabrics have higher bursting strength. Pilling and abrasion resistances are the highest for also 48/52% cotton/modal fabric. For skin friction which is crucial for socks within shoes, rotor viscose, 48/52% cotton/modal and micromodal have smoother surfaces than modal and Tencel® for both kinetic and static friction coefficients. It means that these materials create less injuries on sweaty skin during an activity. It is thought that, results obtained for cotton and different regenerated cellulosic fabrics are valid also for all next-to-skin garments.

Keywords: Skin friction, socks, fiber type, cellulosic, pilling, abrasion, bursting strength.

ÖZET

Pamuk (penye, karde, rotor) ve rejenere selüloz liflerinden (viskon, bambu, modal, mikromodal, Tencel®, Tencel LF® ve bazı karışımlar) üretilen suprem kumaşların deriyle sürtünme ve bazı performans özellikleri sürtünme, patlama mukavemeti, aşınma ve boncuklanma direnci ölçümleriyle belirlenmiştir. Hammadde ve iplik parametreleri günlük veya fonksiyonel bir çorap göz önünde bulundurularak seçilmiştir. Elde edilen sonuçlara göre, en yüksek patlama mukavemeti pamuk, % 48/52 pamuk/modal ve Tencel® kumaşlarda elde edilmiştir. Boncuklanma ve aşınma dirençleri de % 48/52 pamuk/modal kumaşta en yüksek tespit edilmiştir. Ayakkabı içerisindeki çorap açısından hayati bir özellik olan deriyle sürtünme özellikleri incelendiğinde; rotor viskon, % 48/52 pamuk/modal ve mikromodal kumaşların statik ve kinetik sürtünme katsayılarının diğer hammaddelere göre düşük olduğu, dolayısıyla daha pürüzsüz kumaş yüzeyleri oluşturdukları tespit edilmiştir. Buradan, bu hammaddelerin herhangi bir aktivite sırasında terli deride daha az hasar oluşturacağı belirtilebilir. Bu çalışmayla pamuk ve farklı rejenere selüloz lifleri için elde edilen sonuçların deriyle temas halindeki diğer giysi grupları için de fikir verebileceği düşünülmektedir.

Anahtar Kelimeler: Deri sürtünmesi, çorap, lif tipi, selülozik, boncuklanma, aşınma, patlama mukavemeti.

Corresponding Author: Sibel Kaplan, sibelkaplan@sdu.edu.tr

1. INTRODUCTION

Besides thermal comfort, skin-fabric interactions are also important for foot health and comfort. As it is difficult to enable dryness within foot clothing system, deformations as a result of skin-fabric friction due to cyclic relative movements may result injuries, friction blisters which are more problematic for sports and diabetic people. The mechanoreceptors of the skin are very important in the assessment of comfort, because they relay information about fabric roughness and contact forces between skin and fabrics [1]. During walking or running, besides cyclic pressure, friction and shear forces acting on the skin under increased moisture and temperature levels within sports shoes are the main reasons of foot blisters [2]. Skin-fabric interactions are generally evaluated by subjective tests [1, 3-5] combined with fabric surface and skin analyses. There are several objective methods for fabric surface analysis. Friction coefficients between socks fabrics and synthetic

skin were measured using a Textile Friction Analyzer (TFA), and study results were correlated well to preferences of participants obtained by subjective tests [6]. Moreover, a widely used approach is the horizontal platform method, which involves pulling a weighted sled across a fabric adhered to a platform attached to the lower jaw of a tensile tester [1, 6]. Kenins [7] measured static and kinetic friction forces arise during skin contact of fabrics produced from different materials and possess different mechanical and chemical finishing treatments by using different normal forces on dry and wet skin (finger and forearm). He concluded that friction force is more related to wetness of the skin than material or finishing treatment of the fabric. Importance of skin wetness on skin-fabric relation was emphasized in another study [5]. Baussan et al. [2, 8] investigated friction characteristics of cotton athletic socks having different weaves by determining mechanical contacts between foot, sock and shoe during running. They developed a reciprocating linear tribometer to simulate friction contacts between skin and sport socks and created a model. Bogerd et al. [9] measured skin friction between the posterior surface of the calcaneus and a glass plate during wear trials conducted on military members. Van Amber [6] studied effects of fiber type (fine wool, mid-micron wool, acrylic), yarn type (high twist, low twist, single) and fabric structure (single jersey, half-terry, terry) on friction between sock fabrics and a synthetic skin using the horizontal platform method. The effect of weight of a hypothetical wearer and moisture content of the sock fabric were also investigated. Effect of fabric moisture content on friction characteristics were investigated in another study [10] by using FRICTORQ on cotton and cotton/elastane knitted fabrics. Studies about effects of socks on friction blisters on the feet suggest that, the establishment of a movement interface either within the sock itself or between the layers of a sock system will prevent skin injury. Reducing the friction force on skin surface depends upon the fiber composition of the sock, where synthetic fibers appear to work best [11].

There is a growing demand on comfortable and environmentally friendly materials for clothing. Cotton and regenerated cellulosic fibers are common materials used for next to skin garments such as socks, underwear and sports clothing. The reason of their increasing attention is the continuous improvement of their inherent qualities and introduction of new brands in the market like Tencel. Regenerated cellulosic fibers maintain their reputation for softness, strength and good appearance provided to apparel products [12]. Besides comfort, there are also studies in the literature investigating mechanical and performance characteristics of the cellulosic fibers in knitted fabric form. Structural and mechanical performances of knitted fabrics produced from viscose, modal and lyocell were investigated and found out that second and third generation cellulosic fibers have higher bursting strength values due to a higher degree of crystallinity and molecular orientation compared to bamboo and viscose rayon fibers [13-14] as expected. In a study about performances of socks produced from regenerated fibers [15], modal and viscose had similar bursting strength behaviors while strength of bamboo is lower. In other studies about mechanical performance characteristics or cellulosic knitted fabrics, pilling tendency of viscose fiber is found higher than other regenerated fibers [16]. Abrasion was found the highest for lyocell and the lowest for bamboo [17] and modal [14] while in another study, abrasion is maximum for micromodal [15].

The aim of this study is to investigate skin-fabric friction and some other performance characteristics of socks fabrics produced from cotton and regenerated cellulosic fibers by surface friction, bursting strength, pilling and abrasion resistance tests. Tenacity of the regenerated cellulosic fibers have been the main point during production processes but as far as we know, friction characteristics have not been investigated sufficiently. Moreover, relationships among fabric mechanical properties produced from cellulosic yarn were also studied.

2. MATERIAL AND METHODS

2.1. Material

Single jersey fabrics were produced from Ne 30 cellulosic yarns consisting of ring and OE-rotor yarns having identical twist coefficients (α_e = 3.59-3.96). Yarn evenness and tenacity parameters can be seen in Table 1. As can be seen, material group consists of cellulosic fibers; namely cotton, as a natural type, first, second and third generation regenerated cellulosic fibers and their blends. Cotton and viscose include both ring and OE-rotor spun forms. As it is known, viscose is the first generation regenerated cellulosic fiber and because of their mechanical insufficiency in wet form, modal and lyocell fibers were introduced as second and third generation cellulosic fibers. Besides higher wet strength and structural features between viscose and lyocell [18] modal fibers are twice as soft as cotton and are able to withstand repeated wash and dry cycles compared to cotton. Tencel® lyocell fibers are derived from sustainable wood sources and sustainably managed plantations which also enable formation of very long exclusive crystalline arrangement of its cellulose units which are extremely greatly oriented in the longitudinal axis of the fiber [12]. Tencel® fibers exhibit a circular cross section, smooth surface area giving fabrics a soft feel and ensuring comfort for sensitive skin. Both modal and Tencel absorb moisture more efficiently than cotton which is a property supporting body's natural thermal regulating mechanism, keeping skin feeling pleasantly cool and dry [19]. An important feature of lyocell fibers is their propensity of fibrillation due to their high degree of polymer chain orientation and lack of lateral cohesion. Fibrillation is sometimes an advantage such as for creating 'peach skin' but it is not always a desirable property. Cross-linked versions of lyocell such as Tencel A100® and Tencel LF® (low fibrillation) were produced. Tencel LF® is more suitable for blending with cotton as a result of mercerization ability and closer dyeability to that of cotton [17]. Bamboo is also a regenerated cellulosic eco-friendly fiber which has some distinctive properties such as natural anti-bacterial performance and breathability [12].

2.2. Methods

Fabrics and socks were knitted by a Lonati/L462K machine having 3 ½" diameter, 144 needles and a speed of 250 rpm. To investigate the performances of cellulosic fibers, undyed yarns were knitted without elastane. All fabrics were washed according to TS EN ISO 6330:2012 standard in a in a Wascator FOM71 CLS washing machine (James Heal and Co Ltd., Halifax, UK). All tests were conducted under standard atmospheric conditions (20 ± 2 °C, 65±2% RH).

Table 1. Yarn properties of the fabrics

Fabric Code	Material	CVm (%)	IPI Imperfections (Thin+Thick+Neps)	Hairiness (H)	Tenacity (cN/tex)	Breaking Elongation (%)	Work of Rupture (cN.cm)
1	100% Combed cotton	12.26	332.6	4.62	18.99	5.74	577.0
2	100% Carded cotton	14.76	1086.0	5.14	17.96	5.11	493.1
5	100% OE.-Rotor viscose	13.52	678.0	4.05	13.90	12.48	1109.0
6	100% Bamboo	12.21	205.8	5.66	18.05	14.81	1668.0
7	70%/30% Bamboo/ Cotton	13.00	354.0	5.68	13.14	8.24	746.6
8	100% Modal	11.67	104.2	6.46	27.39	11.03	1770.0
9	48%/52% Cotton/Modal	11.45	146.2	6.07	16.53	5.55	551.8
10	100% Micromodal	11.49	81.4	6.07	27.39	10.83	1715.0
11	100% Tencel®	11.38 [0.44]	180.0	6.89	25.39	8.24	1255.0
12	100 % Tencel LF®	11.65	280.0	7.09	25.29	8.10	1237.0

*: Yarn properties belonging to 100% OE-rotor cotton (3) and 100% viscose (4) yarns could not be tested because of sample shortage

Yarn evenness and breaking strength characteristics were tested by Uster Tester 4-S and Uster Tensojet devices in turn. Weight values of the fabrics were determined according to TS 251 and thickness values were measured according to TS 7128 EN ISO 5084 with 5 g/cm^2 pressure. Bursting strength tests were carried out according to ISO 13938-2. Bursting strength (Pa) and elongation occur while bursting (bursting distance) were measured. Pilling and abrasion resistance (weight losses of the samples were recorded for 20.000 revolutions) tests were conducted according to TS EN ISO 12947-2 and TS EN ISO 12945-2 standards respectively by a Martindale Pilling and Abrasion Tester (James H. Heal & Co Ltd., USA).

Friction coefficients of the sock fabrics were calculated by friction force measurements conducted according to ASTM D 1894 by a Lloyd LR5K plus (Lloyd Instruments, Inc., USA) tensile strength tester. Static and kinetic friction forces were obtained for both course and wale directions as a result of movement of a sled (6x7 cm) on a platform covered with the socks fabric inner side up with a speed of 25 mm/min. Sled was covered with a membrane simulating artificial skin and a normal force of 4.72 g/cm^2 was applied during tests. The coefficient of friction μ is defined as the ratio between the frictional force *F* and the applied normal load *N*: $\mu = F/N$ [1, 6, 20-21].

SPSS 21.0 Statistics Software (SPSS Inc. USA) was used for Analysis of variance (ANOVA) test. Duncan and Student Newman Keuls (SNK) tests were used to examine significant differences among measured parameters of the cellulosic socks. A value of p 0.05 indicated statistical significance. Correlation analysis was conducted to determine relationships among physical and mechanical parameters.

3. RESULTS & DISCUSSIONS

As can be seen in Table 2, weight values of the single jersey fabrics ranged between 140-175 g/m^2 and thickness values ranged between 0.70-1.10 mm. Both properties have significant differences according to statistical analyses. Significant changes were marked with letters on Table 2; different letters showing statistical significance. It is thought that weight and thickness differences may be sourced from fiber density and rigidity variations of the fibers. Cotton fabrics have significantly higher thickness values than regenerated cellulosic fabrics. Bamboo (6), modal (8), micromodal (10) and Tencel® (11) have identical and the lowest thickness values.

Fabric mechanical properties are compiled in Table 3. Significant changes were marked with letters on Table 3.

According to bursting strength results, combed cotton fabric (1) has significantly ($p<0.05$) higher bursting strength values than the other fabrics (Figure 1). Carded cotton (2), cotton/modal blend (9), Tencel® (11) and Tencel LF® (12) fabrics having statistically identical performances come after. Fabrics produced from viscose ring and rotor (4 and 5) and bamboo ring (6) yarns have statistically identical and the lowest bursting strength values, which is a result in harmony with preceding studies [13, 15]. Bursting strength values did not have the same tendency with yarn breaking strength values that, while combed cotton (1), carded cotton (2) and cotton modal blend (9) yarns have lower breaking strength values (Table 1) than modal and Tencel, they have identical bursting strength values with Tencel fabrics (11 and 12). This result does not confirm a preceding study result [22] that reported significant relationships between yarn and fabric strength values. We can conclude that transfer of strength from yarn to fabric is better for cotton which may be related to its rougher structure resisting multi-directional deformation of bursting. When yarn spinning techniques are considered, while rotor and ring spininng techniques did not created significant changes for viscose fabric (4 and 5), it created significant differences for cotton and rotor yarn fabric (3) had lower bursting strength values than combed and carded cotton yarn fabrics (1 and 2).

Modal and viscose fabrics have identical strength values in a preceding study [15] but in this study, strength values can be ranked according to the generation of the regenerated cellulosic materials; Tencel fabrics (11 and 12) having the maximum values followed by modal and micromodal (they have identical performances),bamboo and viscose have the minimum bursting strength values. Cotton increased bursting strength values when blended with bamboo (7) and modal (9) as expected.

Table 2. Physical properties of the cellulosic fabrics

Fabric Code	*Material*	*Weight* (g/m^2) [S.D.]	*Thickness* (*mm*) [S.D.]	*Fabric Density* (g/cm^3)	*Pilling Resistance*
1	100% Combed cotton	165.9^{bc} [2.68]	1.05^{b} [0.02]	0.16	2-3
2	100% Carded cotton	175.48^{a} [7.91]	1.10^{a} [0.03]	0.15	2-3
3	100% OE.-Rotor cotton	161.08^{bc} [6.26]	1.02^{c} [0.01]	0.16	2-3
4	100% Viscose	147.95^{de} [4.28]	0.82^{e} [0.02]	0.19	2-3
5	100% OE.-Rotor viscose	175.74^{a} [4.08]	0.81^{ef} [0.04]	0.22	1-2
6	100% Bamboo	155.68^{d} [2.41]	0.79^{fg} [0.03]	0.20	3-4
7	70%/30% Bamboo/Cotton	168.08^{ab} [3.21]	0.88^{d} [0.04]	0.19	3-4
8	100% Modal	147.38^{de} [7.17]	0.74^{g} [0.02]	0.23	2-3
9	48%/52% Cotton/Modal	153.66^{d} [3.79]	0.90^{d} [0.03]	0.18	4-5
10	100% Micromodal	135.96^{f} [4.10]	0.70^{g} [0.02]	0.21	2-3
11	100% Tencel ®	142.90^{ef} [1.89]	0.79^{fg} [0.04]	0.18	1-2
12	100% Tencel LF®	140.00^{ef} [2.88]	0.83^{e} [0.05]	0.19	1-2

Letters on values show statistical significance of the results ($p < 0.05$), same letters having identical properties.

Table 3. Mechanical properties of the cellulosic fabrics

Fabric Code	Material	Bursting Strength (kPa) [S.D.]*	Bursting Distance (mm) [S.D.]*	Weight Loss (%) [S.D.]*	Friction Coefficients [S.D.]*			
					Wale Direction [S.D.]*		Course Diection [S.S.]*	
					μ_k	μ_s	μ_k	μ_s
1	100% Combed cotton	288.52^{a} [25.55]	43.66^{a} [0.68]	5.31^{bc} [0.55]	0.29^{bcd} [0.02]	0.44^{a} [0.06]	0.23 [0.03]	0.36^{bcd} [0.02]
2	100% Carded cotton	264.84^{b} [18.18]	43.04^{a} [1.28]	5.34^{bc} [1.14]	0.32^{bc} [0.01]	0.46^{a} [0.07]	0.19 [0.04]	0.35^{ef} [0.03]
3	100% OE.-Rotor cotton	193.2^{d} [12.52]	42.72^{a} [1.48]	6.67^{abc} [1.49]	0.26^{cde} [0.05]	0.37^{a} [0.04]	0.26 [0.05]	0.37^{abcd} [0.04]
4	100% Viscose	151.6^{e} [15.82]	37.18^{b} [0.52]	8.14^{a} [1.36]	0.21^{ef} [0.01]	0.35^{a} [0.04]	0.21 [0.02]	0.32^{cd} [0.04]
5	100% OE.-Rotor viscose	150.66^{e} [8.59]	38.08^{b} [1.02]	5.33^{bc} [0.76]	0.21^{ef} [0.00]	0.30^{a} [0.04]	0.10 [0.007]	0.20^{f} [0.03]
6	100% Bamboo	162.84^{e} [10.85]	34.56^{c} [1.02]	3.91^{c} [0.67]	0.39^{a} [0.05]	0.37^{a} [0.24]	0.31 [0.13]	0.45^{ab} [0.11]
7	70%/30% Bamboo/Cotton	190.9^{d} [21.04]	38.64^{b} [1.00]	3.76^{c} [0.52]	0.25^{de} [0.02]	0.36^{a} [0.02]	0.31 [0.03]	0.44^{abc} [0.03]
8	100% Modal	219.16^{c} [19.84]	31.64^{d} [1.02]	4.32^{c} [2.01]	0.34^{b} [0.04]	0.35^{a} [0.20]	0.30 [0.02]	0.43^{abc} [0.02]
9	48%/52% Cotton/Modal	261.16^{b} [26.24]	37.56^{b} [1.07]	1.54^{d} [1.31]	0.28^{bcd} [0.02]	0.40^{a} [0.09]	0.19 [0.02]	0.31^{ef} [0.04]
10	100% Micromodal	195.78^{d} [1.90]	28.56^{e} [1.17]	4.07^{c} [0.95]	0.18^{f} [0.04]	0.32^{a} [0.05]	0.21 [0.02]	0.35^{cd} [0.04]
11	100% Tencel ®	265.28^{b} [15.72]	34.48^{c} [1.65]	3.98^{c} [1.31]	0.32^{bcd} [0.005]	0.50^{a} [0.06]	0.35 [0.06]	0.48^{a} [0.09]
12	100% Tencel LF®	244.13^{b} [5.85]	34.05^{c} [1.39]	5.01^{bc} [2.01]	0.32^{bcd} [0.005]	0.51^{a} [0.03]	0.24 [0.04]	0.42^{bcd} [0.07]

Letters on values show statistical significance of the results ($p < 0.05$), same letters having identical properties.

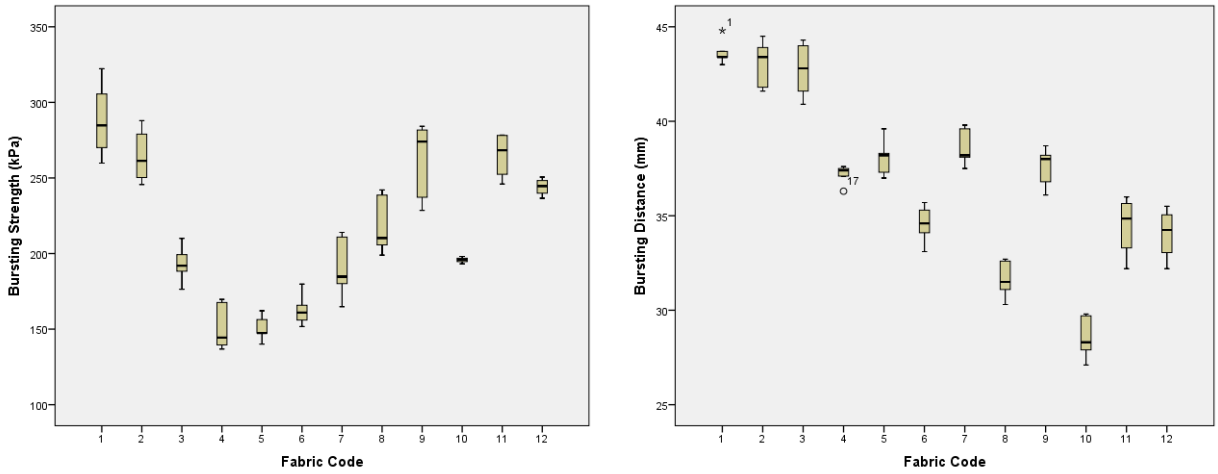


Figure 1. Bursting strength and distance values of cellulosic fabrics *1: 100% Combed cotton, 2: 100% Carded cotton, 3 100% OE.-Rotor cotton, 4: 100% Viscose, 5: 100% OE.-Rotor viscose, 6: 100% Bamboo, 7: 70/30% Bamboo/Cotton, 8: 100% Modal, 9: 48/52% Cotton/Modal, 10: 100% Micromodal, 11: 100% Tencel ®, 12: 100% Tencel LF®*

Bursting distance values showing the elongation of the fabrics while deformation are the maximum for cotton fabrics (1, 2 and 3) and minimum for micromodal fabric (10) as can be seen in Figure 1. Yarn spining technique does not have any influence on elongation values of fabrics for both cotton and viscose. Modal fabric (8) is significantly more eleastic than micromodal fabric. Tencel® (11 and 12) and bamboo (6) fabrics have identical and moderate performances. Viscose (4 and 5), cotton blends of bamboo (7) and modal (9) have statistically identical performances and cotton content increased their elongation values. Fabric bursting distance values do not show a similar trend with yarn breaking elongation values that while cotton yarns have the minimum elongation values (Table 1), their bursting distances are the maximum in fabric form. Higher elongation values of viscose and bamboo yarns which comes from their inherent structures [13] did not create significant differences in fabric form.

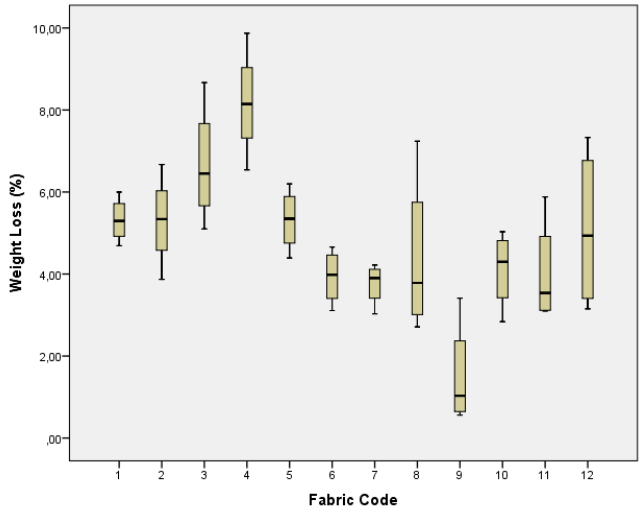
Pilling resistance evaluation results are given in Table 2. Minimum pilling tendency (4-5) was obtained for 48%/52% cotton/modal fabric (9). 100% bamboo (6) and 70%/30% cotton/bamboo fabric (7) had also acceptable pilling performances (3-4). OE-rotor viscose (5) and Tencel® fabrics (11 and 12) had the worst pilling resistance performances that they were evaluated with 1-2 in EMPA scale. Lower pilling resistance of Tencel® can be explained by the fibrillation occuring as a result of mechanical abrasion in wet condition. Standard washing and drying applied before tests might have manipulated pill formation. Moreover, fuzz was mainly generated by dry mechanical abrasion applied during pilling test [23]. Although Tencel LF® includes cross links within the structure to prevent fibrillation [24], its pilling resistance is not superior than standard Tencel® fabric (11). Performances of other fabrics are between the mentioned ones (3-4). The worst performance of viscose (5) was confirmed by preceding studies [16, 22].

Weight loss values after 20.000 cycles of abrasion test are compiled in Figure 2. According to the statistical analysis results, minimum weight loss was observed for 48%/52% cotton/modal fabric (9). Viscose ring yarn fabric (4) lost the highest amount of fiber during abrasion. Fibres with high elongation, elastic recovery and work of rupture have a good ability to withstand repeated distortion; hence a good abrasion resistance [25]. For viscose, fiber cross section and lower fiber strength might have caused high material loss. Abrasion resistances of other fabrics are statistically identical. Effect of yarn spinning technique on abrasion resistance seems insignificant for cotton but fabric produced from ring yarn was abraded more for viscose fabric. This result may be sourced from wrapper fibers on rotor yarn surface increasing abrasion resistance. Tencel LF® yarn fabric (12) having crosslinks within its structure for decreasing fibrillation, hence abrasion did not create significant enhancement when compared with standard Tencel® yarn fabric (11). In a preceding study [17], abrasion resistance of standart Tencel® fabric was the highest and better than bamboo but they had identical performances in this study.

Friction coefficients of the cellulosic fabrics in wale direction can be seen in Figure 3. Differences among static friction coefficients are not statistically significant for wales direction ($p > 0.05$). This result does not confirm a preceding study [6] result stating that the most important effect of fiber was on the static frictional force. According to kinetic friction coefficients, bamboo fabric (6) had the highest value, meaning the roughest surface. Viscose (4 and 5) and micromodal (10) yarn fabrics are the ones having the smoothest surfaces among all cellulosic fabrics. From this result, it can be concluded that fiber cross section do not have an influence on surface friction characteristics. Micromodal yarn fabric (10) have a smoother surface than modal yarn fabric (8) and this result may be attributed to lower diameter decreasing surface roughness and lower bending rigidity of the fibers. Among cotton fabrics, rotor yarn fabric (3) and combed yarn fabric (1) have significantly smoother surfaces than carded yarn fabric (2) and all cotton fabrics have rougher surfaces than viscose fabrics because of their higher fiber bending rigidities. The performances of cotton yarn fabrics are similar with second and third generation regenerated cellulosic fibers having greater degree of crystallinity. Yarn spinning technology does not have an influence on friction characteristics for viscose fabrics (4 and 5). Modal yarn fabric (8) and 48%/52%

cotton/modal yarn fabric (9) have significantly rougher surfaces when compared with viscose fabrics (4 and 5) but they have identical performances with Tencel® yarn fabrics (11 and 12) for kinetic friction coefficients in wale direction.

Static and kinetic friction coefficients calculated for course direction both have statistical significances among fabrics ($p<0.05$). According to static friction coefficient results, viscose rotor yarn fabric (5) had significantly smoother surface than the other fabrics (Figure 4). Bamboo (6), its blend with cotton (7), modal (8) and Tencel® (11, 12) yarn fabrics have significantly rougher surfaces than the other fabrics. Performances of modal and Tencel are in harmony with the results in wale direction and can be explained with the higher crystallinity of the fibers and fibrils if exist enabling higher bending rigidity. Cotton yarn fabrics produced by different spinning techniques have statistically identical performances but rotor yarn fabric has smoother surface than ring yarn fabric (4) in course direction. Kinetic coefficients in course direction have a similar trend that viscose has the smoothest surface followed by modal and Tencel® fabrics. Different from results in wale direction, cotton yarn fabrics (1, 2 and 3) and cotton/modal blend (9) have similar performances with modal. Micromodal and modal yarn fabrics have similar performances for both static and kinetic friction coefficients. Tencel LF® yarn has a smoother surface than standard Tencel® for kinetic coefficent which was not the case for other coefficients. No relationship was found between yarn hairiness and surface friction characteristics.



1: 100% Combed cotton
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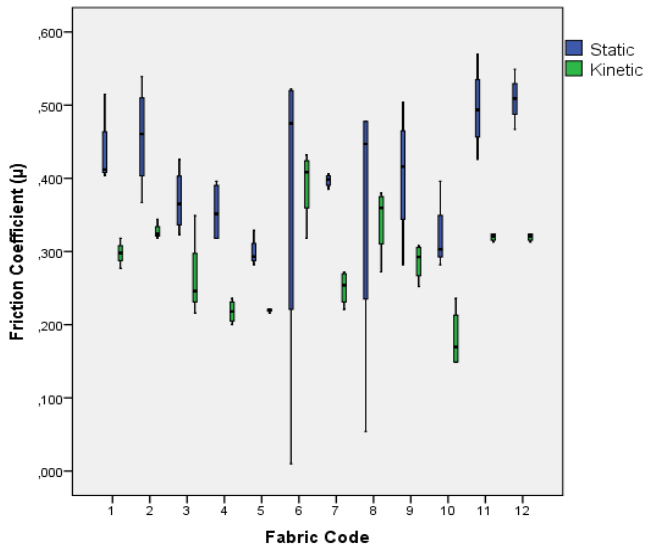
3 100% OE.-Rotor cotton
4: 100% Viscose
5: 100% OE.-Rotor viscose
6: 100% Bamboo
7: 70/30% Bamboo/Cotton
8: 100% Modal
9: 48/52% Cotton/Modal
10: 100% Micromodal
11: 100% Tencel ®
12: 100% Tencel LF®

Figure 2. Boxplot diagram of weight loss values as a result of abrasion



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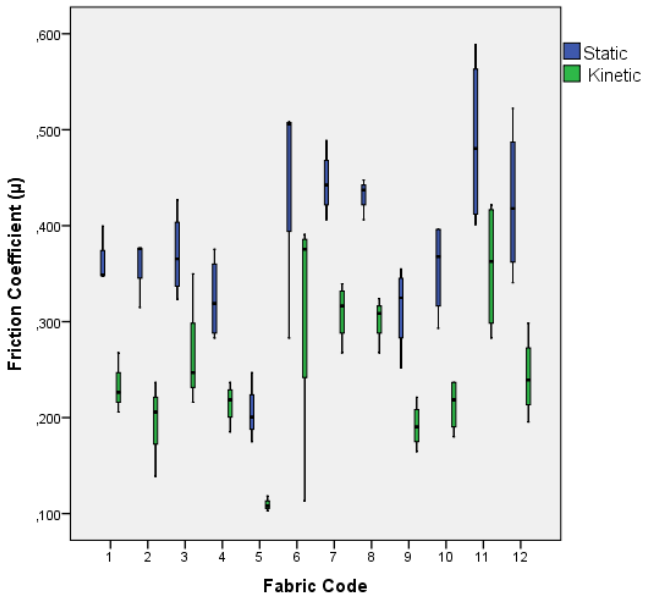
12: 100% Tencel LF®

Figure 3. Friction coefficients in wale direction



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9: 48/52% Cotton/Modal
10: 100% Micromodal
11: 100% Tencel ®
12: 100% Tencel LF®

Figure 4. Friction coefficients in course direction

Significant relationships obtained for yarn and fabric characteristics are summarised in Table 3. Thickness, weight and bursting strength are significantly correlated with each other as expected. Fabric weight is correlated with friction coefficients in course direction; as the fabric becomes heavier and denser, its surface friction decreases, meaning a smoother surface. Weight values are also negatively correlated with yarn breaking elongation, which may be related to the density of the yarn and fiber. It is though that, as the crystalline region of the fiber decreases, yarn becomes more elastic and weight of the fabric decreases. It is interesting to note that bursting strength is negatively correlated with yarn elongation and work of rupture. Higher energy needed for yarn rupture did not contribute the multi-directional deformation of fabric during bursting. Friction coefficients in course and wale directions are correlated with each other and it was observed that differentiation capacity of friction coefficients in course direction is better as they bring forth differences among fabrics more. Thickness has negative relationships with yarn hairiness and yarn strength parameters (elongation and work of rupture). This result may be attributed to higher twist values causing denser yarn structure, higher strength and lower fabric thickness values.

CONCLUSIONS

Skin-fabric friction and other performance characteristics of some socks fabrics produced from cotton and regenerated cellulosic fibers were investigated by surface friction, bursting strength, pilling and abrasion resistance tests. According to the results, cotton and its blend with modal had better performances for bursting strength and cotton/modal had better performances for pilling and abrasion resistances. Besides these performance characteristics, skin-fabric friction is lower for viscose among cellulosic fibers, enabling smoother surface for next to skin garments. Relationships among fabric mechanical and surface parameters were also investigated and significant relationships were observed between fabric weight and friction coefficients which may be valuable for producers. Moreover, fabric bursting strength is correlated with not yarn strength but work of rupture. Friction tests on course direction may be suggested for further studies as they show differences among fabrics better than the results on wale direction. Summing up, cotton/modal blend, having minimum to moderate friction coefficients, good strength, pilling and abrasion resistances can be suggested for daily or functional socks as a material. It is thought that, results obtained for cellulosic fibers may give an idea for all clothing types.

Table 3. Correlation coefficients among yarn and fabric characteristics

	Weight	Bursting Strength	KFC*** (Course)	SFC*** (Course)	SFC*** (Wale)	Thickness
Thickness	0.748**	0.519*	-0.667**	-0.518*		
KFC* (Course)	-0.716**					
SFC* (Course)	-0.663**					
KFC (Wale)			0.485*	0.509*	0.761**	
Yarn Hairiness	-0.642**					-0.511*
Yarn Break. Elong	-0.705**	-0.665**	0.622**			-0.782**
Work of Rupt.(Yarn)	-0.861**	-0.484*	0.754**	0.636**		-0.854**

*: Correlation is significant at the 0.05 level
**: Correlation is significant at the 0.01 level
***: KFC: Kinetic friction coefficient, SFC: is significant at the 0.05 level

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(REFEREED RESEARCH)

PREPERATION AND ANTIBACTERIAL INVESTIGATION OF POLYCAPROLACTONE/CHITOSAN NANO/MICRO FIBERS BY USING DIFFERENT SOLVENT SYSTEMS

FARKLI SOLVENT SİSTEMLERİ KULLANILARAK POLİKAPROLAKTON/KİTOSAN NANO/MİKRO LİFLERİN ÜRETİMİ VE ANTİBAKTERİYEL ÖZELLİKLERİNİN İNCELENMESİ

Omer Firat TURSUCULAR[1], Idris CERKEZ[2], Mehmet ORHAN[1], Yakup AYKUT[1]

[1]Department of Textile Engineering, Engineering Faculty, Uludag University, 16059, Nilüfer, Bursa, Turkey
[2]Department of Fiber and Polymer Engineering, Bursa Technical University, Yildirim, Bursa, Turkey

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ABSTRACT

Chitosan (CHI) blended polycaprolactone (PCL) nano/micro fibers were prepared with different CHI content via electrospinning procedure. Two different solvents, acetone and formic acid (FA) were used to dissolve and blend the polymers before electrospinning process. Effect of the solvent on the electrospinability of the blend, final nano/micro fiber morphologies, chemical and thermal properties and antibacterial activities were investigated with SEM, FTIR, DSC, and ASTM 2149 01 Standard Dynamic Contact Conditions. The results revealed that chitosan particles were encapsulated in the as-spun PCL fibers with using acetone as the solvent resulted in reduced antibacterial activities Contrarily, when FA is used as the solvent, CHI and PCL were dissolved and blended very well, and enhanced antibacterial activities were obtained from as-spun PCL/CHI nano/micro fibers.

Keywords: Antibacterial materials, electrospinning, chitosan, polycaprolactone, nanofiber

ÖZET

Farklı oranlarda karışımı yapılmış kitosan/polikaprolakton (CHI/PCL) nano/mikro lifler elektroçekim metoduyla üretilmiştir. Elektroçekim çözeltileri aseton ve formik asit (FA) olmak üzere iki farklı solvent kullanılarak hazırlanmıştır. Solvent etkisinin üretilen nano/mikro liflerin morfolojileri, kimyasal ve termal özellikleri ve antibakteriyel aktiviteleri üzerindeki etkileri SEM, FTIR, DSC ve ASTM 2149 01 standardına göre antibakteriyellik analizleri yapılarak ölçülmüştür. Elde edilen sonuçlara göre, solvent olarak aseton kullanıldığında kitosanın partikül olarak PCL lifler tarafından kapsüle edildiği ve liflerin antibakteriyellik özelliklerini düşürdüğü gözlemlenmiştir. Diğer taraftan, solvent olarak formik asit kullanıldığında kitosan ve polikaprolaktonun elektroçekim çözeltisi içerisinde tamamen çözündüğü, kapsülasyonun olmadığı, üretilen ağsı yapı içerisinde hem PCL hemde CHI fibriller yapıda bulunduğu ve elde edilen yapıların daha iyi antibakteriyel özelliklerde oldukları tespit edilmiştir.

Anahtar Kelimeler: Antibakteriyel malzemeler, elektroçekim, kitosan, polikaprolakton, nanolif

Corresponding Author: Yakup Aykut, aykut@uludag.edu.tr

1. Introduction

Development of antibacterial materials has critical importance due to increased number of pandemics and epidemics all over the world [**1**]. Therefore antibacterial functionalization of the surfaces is necessary in majority of the areas including textile materials, food packing and the surfaces where people always touch there. Various antibacterial agents including *N*-halamines, metal ions, quaternary ammonium compounds, synthetic mimics of antibacterial peptides and chitosan are reported in the literature [**2, 3**]. Among these, chitosan draws attention due to its biocompatibility and bioactivity [**4**]. Being one of the most abundant polymer after cellulose, chitosan is a reasonable antibacterial material which suitable to use for various applications including textiles, coatings and packing [**4**].

PCL is widely used in biomedical applications due to its unique biocompatible and biodegradable structure. PCL nanofibers have been developed for use as wound dressings [**5**], biomedical scaffold for tissue engineering [**6, 7**], drug delivery [**8**], vascular grafts [**9**], and water filters [**10**]. All of these aforementioned applications require antibacterial functionalities as they are mainly used in medical environment. In this regard, chitosan was mainly used to fabricate antibacterial PCL membranes by blending chitosan and PCL in a common solvent [**11, 12**]. Along with blend electrospinning, grafting and coaxial electrospinning were also used to develop chitosan grafted PCL

nanoparticles [**13**] and core-shell PCL/CHI nanofibers, respectively. PCL/ CHI nanofibers for liver tissue engineering has been also investigated [**14**].

Electrospinning is a novel technology that helps to produce continues nanofibrous structure from variety of precursors to final structured materials including polymers, ceramics and carbon [**15-17**]. In the procedure, a proper solution is prepared from the precursor and electrospun into nanofibers by applying high voltage electricity. Because of the application of electricity, the solution droplet is elongated until reaching to collector and form nanofiber structure. During the journey of the solution droplet, solvent is evaporated and the dried fibers are collected.

It is well kwon that the solvents used to dissolve PCL for electrospinning affects the surface morphologies, electrospinnability and biocompatibility. This relationship is well studied in the literature [**18**]. Various studies have been reported on PCL/CHI nanofibers with antibacterial activities, solvent effect on the morphologies and the biocidal activities of the PCL/CHI nanofibers has not systematically been investigated yet. In this this study compares morphological, thermal and biocidal properties of the PCL/CHI nano/micro fibers fabricated with blending in formic acid and acetone.

2. Materials and Methods

2.1. Chemicals

For electrospinning solution, Chitosan (Mw: 100,000-300,000) was obtained from Acros Organics and polycaprolactone (Mn:80,000) was received from Sigma Aldrich. Acetone and Formic Acid obtained from Sigma Aldrich were used as the solvent. For antibacterial tests, the mediums for supporting growth and cultivation of microorganism such as Tryptic soy broth (TSB), Nutrient Broth (NB) and Nutrient Agar (NA) were supplied by Becton Dickinson. For preparing buffer solution, $NaH_2PO_4.2H_2O$ (sodium dihydrogen phosphate) and $Na_2HPO_4.12H_2O$ (di-sodium hydrogen phosphate dodecahydrate) were purchased from Sigma Aldrich.

2.2. Electrospinning method

15 wt% of PCL was dissolved in acetone and FA separately by magnetically stirring. Then, a proper amount of chitosan (PCL/CHI: 95/5, 80/20, 50/50) was added in the as-prepared solution and stirred. Final prepared solution was loaded in a plastic syringe fitted with a stainless steel needle (0.508 mm i.d.). A grounded metal collector was placed in front of the needle where polymer solution was fed. Applied voltage and the distance were around 15 kV and 15 cm. Flow rate were around 4 ml/hr for acetone samples and 1 ml/hr for FA samples. Polymer solution droplet in front of the metal needle was ejected from needle to collector plate as a result of the applied voltage. Finally, the solvent was evaporated until the nano/micro fibers reach to the collector and dried nano/micro fibers were collected on the grounded collector plate.

2.3. Nano/micro fibers characterizations

The morphology of nano/micro fibers was explored using a scanning electron microscope (ZEISS EVO 40 with thermionic electron gun) with an acceleration voltage of 20 kV. In order to reduce charging during SEM imaging and getting clear images, samples were placed on a sample holder and coated with gold-palladium in a proper thickness using a BAL-TEC SCD005 sputter coater. Nano/micro fibers were also analyzed with an optic microscope. Attenuated total reflection Fourier transform infrared spectra (ATR-FTIR) of nano/micro fibers were recorded using a Thermo Nicolet iS50 in the wavenumber range of 4,000 to 400 cm^{-1} at room temperature. At least 124 scans were collected to minimize noise. DSC data were obtained using a Perkin Elmer DSC 8000. The experiments were conducted with a heating rate of 10^oC/min under nitrogen atmosphere.

2.4. Antibacterial activity measurements

The antibacterial efficiency was quantitatively evaluated by the ASTM 2149 01 Standard Dynamic Contact Conditions. The samples were tested against Gram-negative bacteria (*Escherichia coli* ATCC 35218). In this test, a homogenous suspension of bacteria was prepared in NB and diluted with buffer solution at pH 7. The standardized concentration of about 10^5 cfu/ml was applied to samples for the antibacterial testing. All the samples were incubated at 37°C and shaken in a wrist-action shaker for 24 hours. The antibacterial activity was expressed in % reduction of the organisms after contact with the test sample at time '0'compared to the number of bacterial cells surviving after contact with the sample after 24 hours. The percentage reduction (R) of bacteria was calculated using following formula: R = 100 (B-A) / B, where, A is the number of bacteria recovered from the inoculated treated test sample in the jar incubated for 24 hours; and B is the number of bacteria recovered from the inoculated treated test sample at '0' contact time. Antibacterial test procedure were adapted to the previous reported papers by Orhan et al. [**19, 20**].

3. Results and Discussions

As a concept in this study, first CHI/PCL solutions were prepared by using acetone and FA separately. Schematic illustration of the preparation, electrospinning process and nanofiber morphologies of the as-spun CHI/PCL nano/micro fibers by using acetone and FA solvents are demonstrated in Figure 1. As seen from SEM and optical microscopy images, the as-spun nano/micro fibers structures were differentiated. When acetone was used as the solvent, CHI particles were encapsulated by PCL nano/micro fibers (Figure 1, A1, A2). On the other hand, when FA was used, ultrafine fibrillar structures were formed among the blended nano/micro fibers (Figure 1, B1, B2). The details of the phenomenon are discussed as following.

3.1. Morphology analysis of as-spun nano/micro fibers

High magnification morphology analyses of fibers were conducted via SEM and given in Figure 2 and 4. Fiber average diamters were determined from SEM images. Average fiber diameters range for fibers prepared from CHI/PCL solutions by using acetone, 1234 nm for pure PLC and increases upto 1648 nm for PCL/CHI (95/5), 1682 nm for PCL/CHI (80/20) and 2832 nm for PCL/CHI (50/50). As seen from Figure 2, increased fiber diameter observed with addition of chitosan into PCL nano/micro fibers. Chitosan particles were encapsulated in the PCL nano/micro fibers when acetone was used as the solvent. The encapsulation was clearer in high concentration chitosan sample (Figure 2D).

In order to support chitosan encapsulation in the ultrafine nano/micro fibers, optical microscopy analysis was also

conducted. As seen in Figure 3, encapsulated chitosan concentration increased with addition of more chitosan in PCL-acetone electrospinning solution, since chitosan particles were not totally dissolved in the solution. Because chitosan particles were encapsulated, fiber breakage was also observed at some points as a result of stress formation along the fibers during electrospinning.

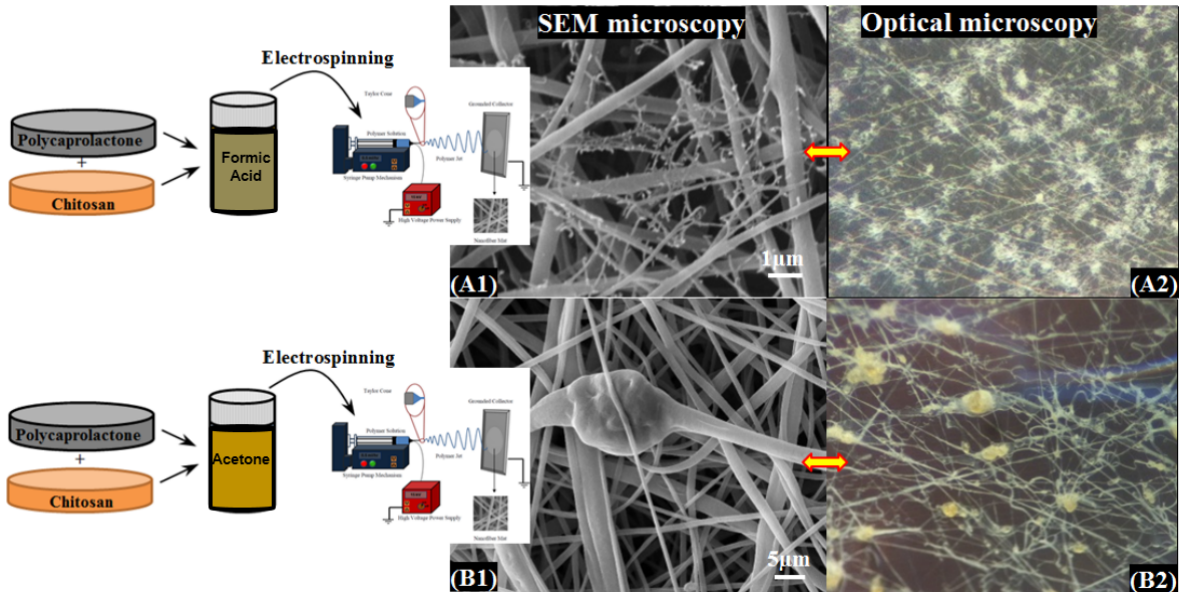

Figure 1. Schematic illustarion of solution preparation, electrospinning process and nanofiber morphologies of as-spun CHI/PCL (50/50) nano/micro fibers by using acetone and FA as solvents. The used magnifications for optical microscopy are 600x.

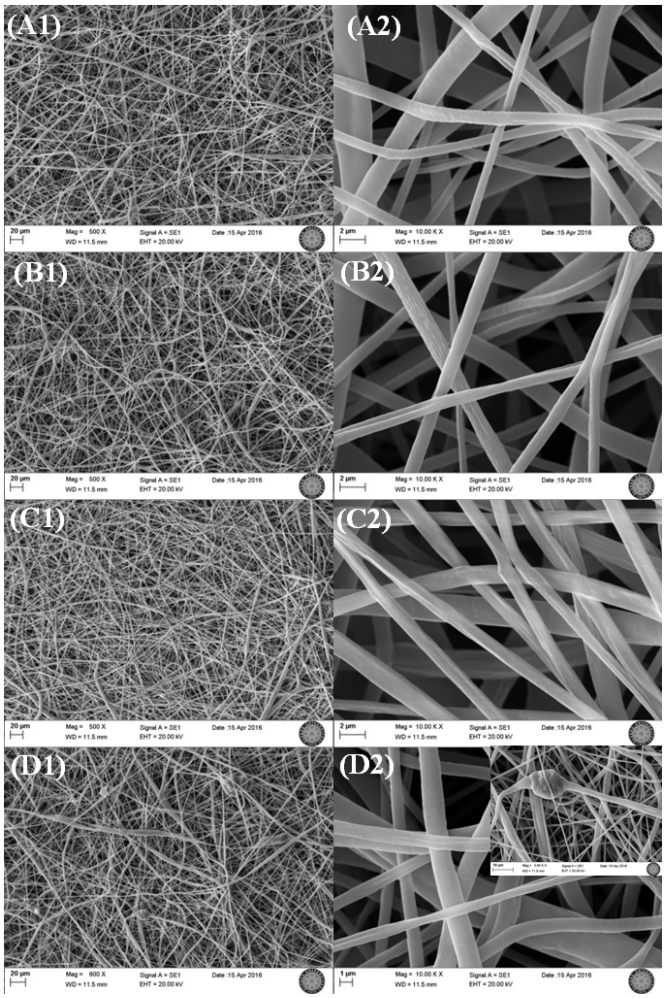

Figure 2. SEM images of acetone solutions of as-electrospun (A1, A2: PCL), (B1, B2: PCL/CHI (95/5)), (C1, C2: PCL/CHI (80/20)) and (D1, D2: PCL/CHI (50/50)) nano/micro fibers.

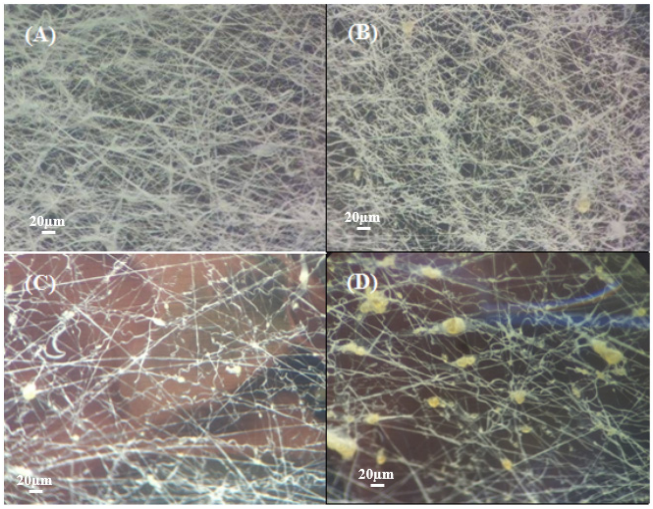

Figure 3. Optic microscope images of acetone solutions of as-electrospun (A: PCL), (B: PCL/CHI (95/5)), (C: PCL/CHI (80/20)) and (D: PCL/CHI (50/50)) nano/micro fibers. These nano/micro fibers are the same as in Figure 2.

Fiber average diamters were determined from SEM images. Except to ultrafine nano fibers, average fiber diameters in the mat for FA solution prepared nano/micro fibers range from 214 nm for pure PLC and decreases to 171 nm for PCL/CHI (95/5) and increases again by increasing CHI contents in nanofiber upto 187 nm for PCL/CHI (80/20) and 446 nm for PCL/CHI (50/50). Ultrafine nano fibrillation was observed in the nanofiber mat with addition of chitosan and this trend increased with increasing chitosan concentration (Figure 4B1-4B4). Similar trend was also reported by Schueren et al. by electrospinning of PCL/CHI NFs using acetic acid/formic acid mix solution **[21]**.

In order to check if there was encapsulation as in the case of PCL/CHI-fa nano/micro fibers, optical microscopy analyses were also conducted for the PCL/CHI-acetone samples. As seen in Figure 5, no chitosan encapsulation was observed. Instead, blended PCL/CHI nano/micro fibers were obtained. This can be proved by looking at the electrospinning solution, since PCL/FA solution was transparent supporting homogeneous solution formation.

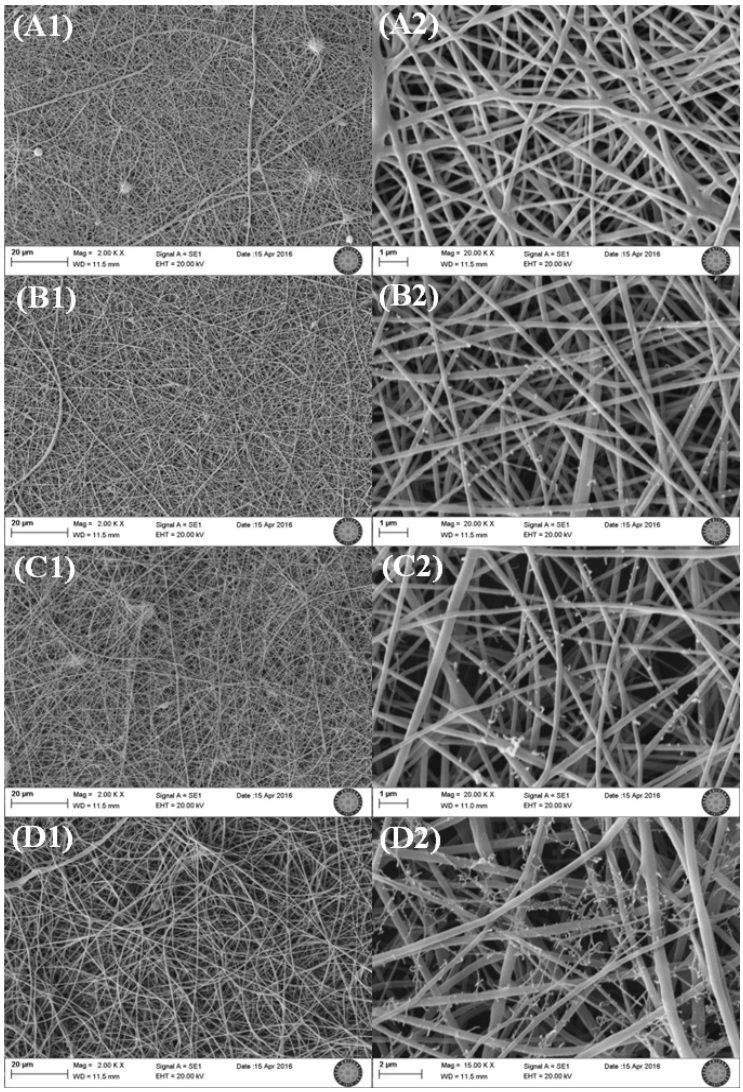

Figure 4. SEM images of FA as-electrospun (A1, A2: PCL), (B1, B2: PCL/CHI (95/5)), (C1, C2: PCL/CHI (80/20)) and (D1, D2: PCL/CHI (50/50)) nano/micro fibers.

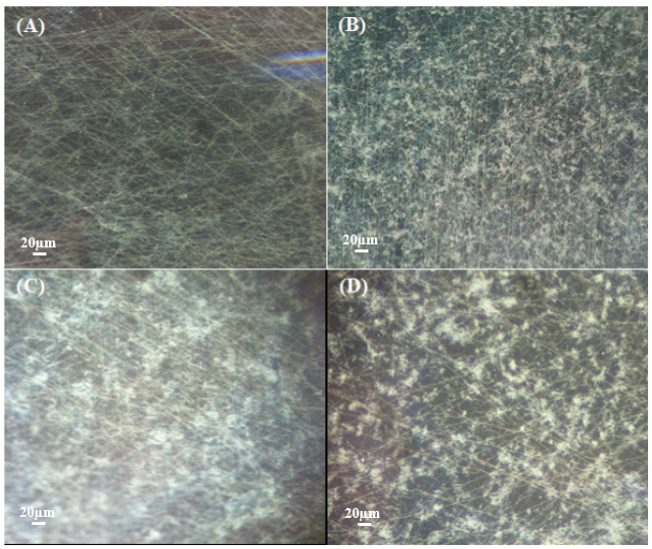

Figure 5. Optic microscope images of FA as-electrospun (A:PCL), (B: PCL/CHI (95/5)), (C: PCL/CHI (80/20)) and (D: PCL/CHI (50/50)) nano/micro fibers. These nano/micro fibers are the same as in Figure 4.

3.2. Chemical analysis via ATR-FTIR

Figure 6A and 6B show FTIR spectra of the produced nano/micro fibers using acetone and FA, respectively.

Along with typical absorption bands of CH_2 stretching at 2943 and 2864 cm^{-1}, the prominent peaks were detected at 1165, 1467 and 1726 cm^{-1} for the neat PCL nano/micro fibers corresponding to the symmetric stretching of C-O-C, asymmetric deformation of CH_3 groups, and stretching bands of ester carbonyl groups, respectively [**22, 23**]. There is no distinctive difference obtained between the neat PCL and PCL-chitosan blends for the samples produced with acetone. On the other hand, new absorption bands were detected at 3356, 1666, and 1569 cm^{-1} for the chitosan blended samples produced with FA. Moreover, the intensities of these new bands increased with increasing chitosan amount. These bands are the characteristics absorptions for chitosan, such that the bands at 3356 cm^{-1} correspond to the N-H vibrational stretching, the bands at 1666 cm^{-1} correspond to the C=O vibrational stretching and the bands at 1569 cm^{-1} correspond to the N-H vibrational bending [**24**]. The reason for not observing any chitosan vibrational bands for the samples produced with acetone is that the chitosan is encapsulated by PCL as evidenced by SEM images. Since only surface functional groups are detected by FTIR with ATR unit, amide group vibrational bands could not be observed for these samples.

3.3. Thermal analysis of as-spun NFs with DSC

As seen from DSC analysis results of the as-spun NFs in Figure 7, all chitosan blended PCL nano/micro fibers showed an endothermic peak around 58 ^{o}C which was associated with the melting temperature of PCL [**22**]. Even though melting point of the acetone sample did not change with addition of chitosan, a slight left shift was observed for FA samples with chitosan addition up to 50/50. This revealed that miscibility of chitosan and PCL was higher in FA then in acetone [**22**]. This was also observed at electrospinning solution that even though solution PCL/CHI in acetone was blurry, solution PCL/CHI in FA was totally transparent. On the other hand SEM images of PCL/CHI NFs also support this result that because of partially solubility in acetone some CHI particles were encapsulated by PCL/CHI composite nano/micro fibers.

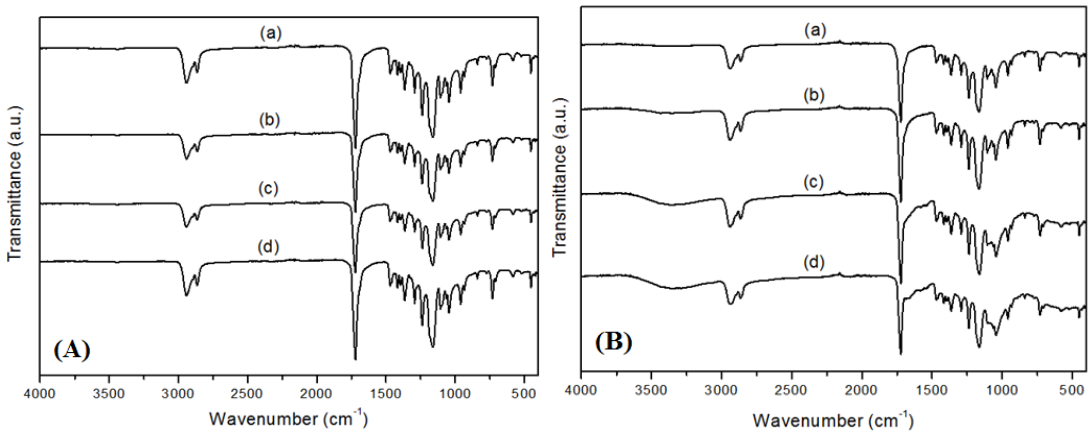

Figure 6. FTIR spectra of PCL/Chitosan nano/micro fibers with different ratio: a) pure PCL nano/micro fibers, b) (PCL/CHI (95/5)), c)(PCL/CHI (80/20)) and d)(PCL/CHI (50/50)) nano/micro fibers. (A): Acetone solution, (B): FA solution.

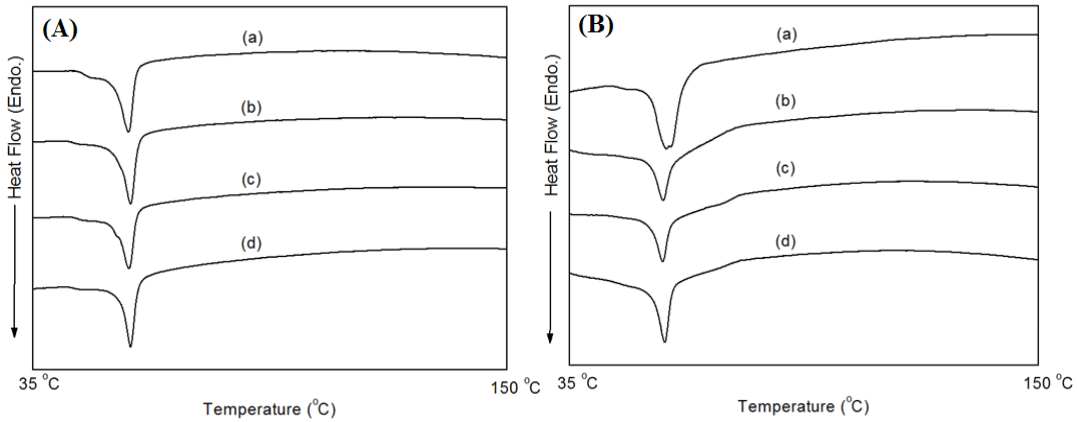

Figure 7. DSC thermo analyses of: a) pure PCL nano/micro fibers, b) (PCL/CHI (95/5)), c)(PCL/CHI (80/20)) and d)(PCL/CHI (50/50)) nano/micro fibers. (A): Acetone solution, (B): FA solution.

3.4. Antibacterial activity measurements

Electrospun PCL/CHI nano/micro fibers produced by using FA and acetone for electrospinning solution preparation results both different nano/micro fibers morphologies and antibacterial activities. In this regards, four different concentration of PCL/CHI nano/micro fibers for both acetone and FA samples were produced and antibacterial activity tests were conducted against *E. coli*, and the results are demonstrated in Table 1.

Chitosan is a biopolymer which represent antibacterial activity [**12, 25, 26**] It was expected that addition of chitosan into PCL exhibit antibacterial activity. According to test results, it was seen that PCL surfaces obtained using both FA and acetone dissolution solvents exhibited no antibacterial activities (-3.23% and -3.23%, respectively) against *E.coli*. After combining CHI at varying ratios in PCL, the antibacterial activities against *E.coli* were enhanced by increasing CHI concentrations for FA and acetone solvents (-94.84% and -35.48%, respectively). It has been demonstrated that the antibacterial activities were dramatically increased (from -3.23% to -94.84% for FA) after adding CHI in PCL, which implies that CHI is a good bactericide. These results are compatible with previous studies for the representation of antibacterial property of chitosan [**12, 25-28**]

Since chitosan particules were encapsulated by PCL in PCL/CHI hybrid nanofiber structure if acetone was used as solvent for electrospinning solution, The acetone solvent system for PCL/CHI showed no sufficient antibacterial activities (from -3.23% to -35.48% for AC) against *E.coli*. PCL/CHI (50-50) surfaces obtained using FA have the best antibacterial activity against *E.coli*. Because CHI encapsulation was not observed at PCL/CHI nano/micro fibers when FA was used as solvent, CHI properly distributed in the mat and helps to promote antibacterial activity.

Table 1. The antibacterial activity results against *E. coli*[a] according to test method ASTM E2149

Sample number	Sample name			The antibacterial activity after 24 h (Bacteria reduction, %)
1	Control sample			No reduction
	Nanofiber	Solvent	Concentration (PCL/CHI)	
2		FA	(100-0)	3.23
3			(95-5)	35.48
4			(80-20)	79.03
5			(50-50)	94.84
6		AC	(100-0)	3.23
7			(95-5)	6.45
8			(80-20)	19.35
9			(50-50)	35.48

[a] The concentration of bacteria was adjusted to 3,10 x10^5 (log 5,49) cfu*/ml for each sample.
* cfu : Colony forming unit
** PCL: Polycaprolactone, CHI: Chitosan, FA: Formic acid, AC: Acetone
Note: The positive values of bacterial reduction (%) demonstrate a decrease in bacterial growth.

CONCLUSIONS

The type of dissolution solvent for electrospun PCL/CHI nano/micro fibers and its effect on nanofiber morphologies and their antibacterial activities were investigated. It was found that when FA was used as a solvent for electrospinning of PCL/CHI polymer blend nano/micro fibers, antibacterial activities considerably increased compared to acetone used as a solvent. This result is believed to be mainly due to more homogenous nanofiber structures were obtained when FA used as solvent. Optical and electron microscope images, and DSC and FTIR analysis also supported that encapsulation of chitosan by PCL nanofiber matrix when acetone electrospinning solution was used resulting in limited contact of chitosan with bacteria thus weakening the antibacterial properties. In conclusion, the FA system showed more potential for the solution electrospinning of antibacterial PCL/CHI polymer blend nano/micro fibers.

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(REFEREED RESEARCH)

PA6/SILVER BLENDS: INVESTIGATION OF MECHANICAL AND ELECTROMAGNETIC SHIELDING BEHAVIOUR OF ELECTROSPUN NANOFIBERS

PA6/GÜMÜŞ KARIŞIMLARI: ELEKTROEĞİRME İLE ÜRETİLMİŞ NANOLİFLERİN MEKANİK VE ELEKTROMANYETİK KALKANLAMA ETKİNLİKLERİNİN ARAŞTIRILMASI

Erhan SANCAK[1], Mustafa Sabri OZEN[1], Ramazan ERDEM[2], Ali Can YILMAZ[3*], Metin YUKSEK[1], Navneet SOIN[4], Tahir SHAH[4]

[1]Marmara University, Technology Faculty, Department of Textile Engineering, Istanbul, TURKEY
[2]Akdeniz University, Serik G-S. Sural Vocational School of Higher Education, Department of Textile Technologies, Antalya, TURKEY
[3]Çukurova University, Adana Vocational School of Higher Education, Department of Motor Vehicles and Transportation Technologies, Adana, TURKEY
[4] Institute for Materials Research and Innovation (IMRI), University of Bolton, Bolton, UK

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ABSTRACT

PA6 nanofibrous membranes containing different amount of silver nanoparticles AgNP (0wt.%, 1wt.% and 2wt.%) were fabricated by a basic electrospinning set up that includes "top to bottom" feeding system. Morphology of the membranes was observed with SEM analysis. Beadless and almost uniform nanofibers were yielded with the average fiber size ranged from 174 nm to 238 nm. Mechanical tests were also carried out in order to explore the tensile and elongation properties of the electrospun membranes, and it was noticed that the increased amount of AgNP in the blends decreased the mechanical performance of the membranes. In addition, fiber size distribution, feeding direction and agglomeration of AgNp were highly influent factors on both morphology and mechanical characteristics of the membranes. Electromagnetic shielding effectiveness (EMSE) of the electrospun nanofibrous membranes was determined according to the ASTM D4935-10 protocol by using coaxial transmission line measurement technique in the frequency range of 15–3000 MHz. It was observed that the membrane containing 2wt.% AgNP performed better EMSE than the others.

Keywords: Electrospinning, EMSE, nanofiber, nano silver, mechanical strength

ÖZET

Farklı oranlarda gümüş nano parçacık (AgNP ağırlıkça %0, %1 ve %2) içeren PA6 nano lif membranlar "üstten alta" beslemeli elektroeğirme ile üretilmiştir. Membranların morfolojileri SEM analiziyle incelenmiştir. Neredeyse eş dağılıma sahip ve ortalama boyutları 174 nm ile 238 nm arası değişen nano lifler elde edilmiştir. Elektroeğirmeyle elde edilen membranların çekme ve uzama özelliklerinin tayini için mekanik testler ayrıca gerçekleştirilmiştir. Yapılan deneylerde karışımlardaki AgNP miktarı arttıkça membranların mekanik performanslarının azaldığı tespit edilmiştir. Bunun yanı sıra, AgNP lif boyut dağılımının, besleme yönünün ve çökelmenin membranların morfolojik ve mekanik karakteristikleri üzerinde büyük etkileri olduğu tespit edilmiştir. Membranların elektromanyetik kalkanlama etkinlikleri (EMKE) ASTM D4935-10 standardı baz alınarak koaksiyel transmisyon ölçüm tekniğine göre 15-3000 MHz aralığında belirlenmiştir. Yapılan deneylerde ağırlıkça %2 AgNP içeren membranın en iyi EMKE gösterdiği sonucuna varılmıştır.

Anahtar Kelimeler: Elektroeğirme, EMKE, nanolif, nanogümüş, mekanik dayanım

Corresponding Author: Ali Can Yılmaz, acyilmaz@cu.edu.tr

INTRODUCTION

Nanofibers and nanofibrous composites have recently gained impetus because of their distinguished properties such as large surface area to volume ratio, highly porous structure with very small pore size and expanded pore interconnectivity, variety of options in surface functionalities and superior mechanical performance compared with the other material forms. Hence, nanofibers ensure various specific usages in many developing application areas including tissue engineering, wound dressings, drug delivery, skin therapy, catalyst and enzyme carriers, filtration, protective equipments, nano-sensors and nano-electronics, composite reinforcement, energy harvest and storage, optics, electromagnetic interference shielding and so on [1–3].

Electrospinning is a very simple and versatile process that enables electrostatic forces to fabricate polymer fibers in submicron range [4]. Preparation of polymer fluid (solution or melt), charging of the fluid, formation of the cone-jet, fining of the instable polymer jet in an electric field and deposition of the fibers on a collecting platform are the characteristic processing steps of electrospinning. Solution properties (viscosity, conductivity, molecular weight, surface tension, polymer concentration, and solvent types and blends), process parameters (intensity of applied electric field, collection distance and feeding rate) and ambient conditions (humidity and temperature of the environment) are influent factors that affect fiber diameter and morphology during electrospinning [5-8].

Potential hazardous effects of electromagnetic interference (EMI) on living creatures as well as on the performance of electronic equipments are common concerns among scientists [9]. The fundamental cause of EMI formation is the undesired electromagnetic emission being radiated or conducted [10].The term “Electromagnetic Shielding Effectiveness (EMSE)” is described as the inducing of EMI by using conductive and magnetic barriers [11]. This effectiveness strongly depends on electromagnetic frequency, thickness of the shield and the distance between the shield and the source. One can compute EMSE in decibels (dB) using Eq. (1):

$$SE = 10\log\frac{Po}{Pt} = 20\log\frac{Eo}{Et}\ 20\log\frac{Ho}{Ht} \qquad (1)$$

where P_0, E_0 and H_0 are the power, the electric and the magnetic field intensities pertaining to the shield, respectively and P_t, E_t and H_t are the factors transmitted through the shield [12].

Though metals are the most effective electromagnetic shielding materials, polymer composites are more likely to be put into use due to their lower cost and weight, corrosion resistance, better thermal stabilities, ease of manufacturing and shaping [13]. Therefore, applying the conductive components such as metallic particles into/onto the textile-based structures (fibers, fabrics, films *etc.*) is the main point in inducing the EMI effect. Several studies have recently been reported in the literature in terms of developing conductive nanostructures by using electrospinning technique. Erdem et. al. electrospun Polyamide 6 nanofibers and sputter coated them with gold and palladium to create functional nanofibrous membranes that possess EMI shielding ability [14]. Kim et. al. used both electrospinning and metal deposition methods to prepare Fe_2O_3 added nanofibrous composites made of Ag-decorated poly(vinyl alcohol) for EMI shielding applications [15]. Chiscan et. al. explored the microwave frequency absorption of PVC/ʎ-Fe_2O_3, PVC/Fe_3O_4, and PVC/$CoFe_2O_4$/CoO nanofibers produced by electrospinning [16]. Fu et. al. utilized two different methods as electrospinning and sol-gel to fabricate highly conductive silvered electrospun silica nanofibers via Poly(dopamine) functionalization [17]. Kim et. al. utilized metal deposition (Cu, Ni, Ag) and electrospinning techniques to obtain nanofiber mats with EMI shielding properties [18].

Unlike the above studies which are complicated and include more than one processing step, in our experimental study, silver nanoparticles (AgNP) were added into the polymer solution directly and the electrospinning process was conducted in newly manufactured equipment which was designed and constructed by our crew. In this test rig, metal particles in the solution are not allowed to precipitate or agglomerate for a while. High electrical conductivity, stretchability, corrosion resistance and thermal stability under wide temperature range make silver one of the most appropriate candidate for metal filler in composite structures [19-23]. Applications on electrical devices using silver nanowires such as electrodes, low temperature sintered conductive adhesives, transparent conductive film, superconductive thick film circuit, microwave absorbing materials and electromagnetic wave absorbing materials have also been carried out [24-30]. Nylon 6 (PA6) is considered as an important class of thermoplastics that has high modulus, good mechanical strength, dimensional stability under even elevated temperatures and chemical resistance against many moderately polar and non-polar organic species [31-33].

EMSE is defined as a specific value for EMI protection. On the other hand, mechanical characteristics are very crucial for the materials regarding manufacturability, lifetime and resistance against external effects. The purpose of this study is to produce AgNP incorporated PA6 nanofibrous membranes by using electrospinning technique and to determine the mechanical characteristics and EMI shielding performances of these membranes.

Experimental Work

2.1.Materials

PA6 (Tecomid®, *d:* 1.49 g/cm^3) was provided by Eurotec (Turkey). Silver nanoparticles (20nm, spherical, metal basis, *d:* 10.5 g/cm^3) was obtained from US Research Nanomaterials, Inc. and formic acid (FA) was purchased from Sigma Aldrich (Germany).

2.2. Preparation and characterization of the polymer solution

Three different solutions (Pure PA6; PA6 with 1 wt.% AgNP; PA6 with 2 wt.% AgNP) were prepared for the experiments. Firstly, PA6 (20 wt.%) was dissolved in FA by utilizing laboratory type magnetic stirrer (Stuart, SB 162) at room temperature for 6 h. Then, AgNP was added to the solutions at the ratios mentioned above. As the nanoparticles added

into the polymer solution, they exhibited a tendency to precipitate after a while due to the clustering activity of the particles. In order to disperse these particles, after stirring, sonication (220V/50-60 Hz) was performed for 1 h in an ultrasound generator (Alexandra Ultrasonic). Viscosities of the solutions were determined with Brookfield Digital Viscosimeter by using S21 type spindle with the rotational speed of 20 rpm. Electrical conductivity of the solutions was also measured with a laboratory type conductivity meter (WTW, Cond3110) under ambient atmosphere.

2.3. Electrospinning process

Electrospinning was carried out in the self-designed laboratory spinning unit (feeding direction is from top to bottom, Figure 1). Matsusada Handy Type (Japan) was used as high voltage power supply unit, and microfluidics syringe pump (NE 1002X) was used for solution feeding. Each solution was placed in a 10 ml syringe and sent to the drum collector (covered with aluminum foil) through a 20-gauge nozzle. The power supply (AC) was set up for a positive voltage of 32 kV. The flow rate of the solution was also adjusted by setting up the syringe pump at 0.40 ml/h. The rotational speed of the drum collector was 100 rpm and its distance was set to be 13 cm away from the nozzle. During the experiments, relative humidity and temperature values ranged from 40% to 45% RH and 28°C to 30°C, respectively.

2.4. Mechanical Testing of Nanofibrous Membranes

Firstly, membrane thicknesses were measured according to TS 128 EN ISO 5084 by using R&B Cloth Thickness Tester (James H. Heal & Co. Ltd.). Then, in order to determine the mechanical properties (tensile strength and elongation) of the Pure PA6, PA6 with 1wt.% and PA6 with 2 wt.% nanofibrous membranes, tensile and recovery tests were performed by utilizing an Instron Machine (Instron4411) at ambient environment (22°C± 3°C and 50% ± 5%RH). The samples were cut into approximately 50 mm × 10 mm (length × width) in both machine and width directions in order to load properly into the uniaxial testing machine. During the experiment, 50N load cell under a cross-head speed of 10 mm/min was applied to the samples. Five repetitions were taken for each sample in order to calculate the average tensile strength and elongation at break values.

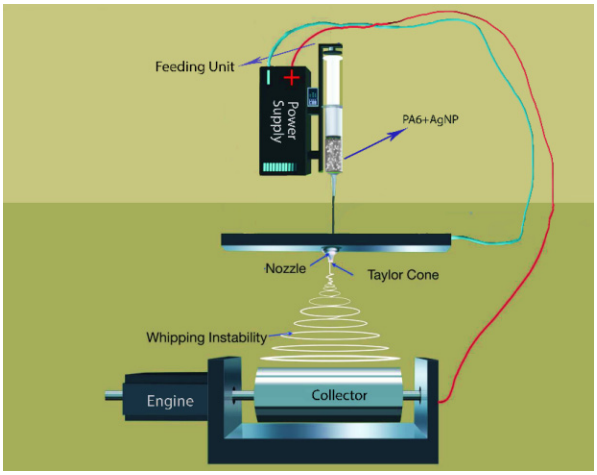

Figure 1. Schematic illustration of electrospinning set up

2.5. Measurement of Electromagnetic Shielding Effectiveness (EMSE)

All nanofibrous membranes were conditioned at 20°C± 2°C temperature and 65% ± 2% relative humidity. Measurements were repeated three times on different areas of the membranes and the average values of the measurements were calculated. A coaxial transmission line method specified in ASTM D4935-10 was used to test the EMSE of the knitted fabrics. The specimen was prepared with a standard test size of various thicknesses. The outer ring of the specimen was 133 mm in diameter. Two specimens were used for the test, one for reference and another for load testing. Various researchers have described the detailed set-up and testing procedure using a plane-wave electromagnetic field in the frequency range of 15MHz–3GHz. A network analyzer (Rohde Schwarz, ZVL) to generate and receive the EM signals and a shielding effectiveness test fixture (Electro-Metrics, Inc., EM-2107A) were used to measure the EMSE, which was measured in decibels (dB) [34,35].

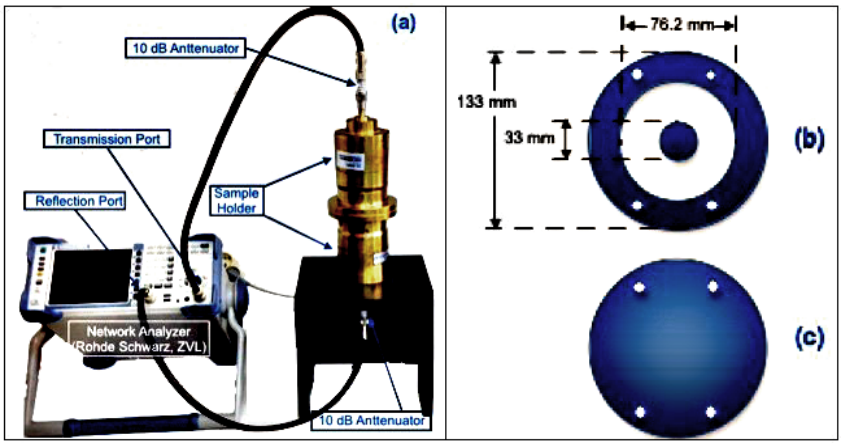

Figure 2. a) Set up of the Electromagnetic Shielding Effectiveness testing apparatus; (b) and (c) Specimen for reference and load respectively.

This standard determined the shielding effectiveness of the fabric (in our case nanofibrous membrane) using the insertion-loss method. The technique involved irradiating a flat, thin sample of the base material with an EM wave over the frequency range of interest, utilizing a coaxial transmission line with an interrupted inner conductor and a flanged outer conductor. A reference measurement for the empty cell was required for the shielding-effectiveness assessment (Figure 2(b)). The reference sample was placed between the flanges in the middle of the cell, covering only the flanges and the inner conductors. A load measurement was performed on a solid disk shape, which had an equal diameter with the flange (Figure 2(c)). The reference and the load measurement were performed on the same material. The shielding effectiveness values were evaluated from Eq. (2), which is the ratio of the incident field to that which passes through the material [34].

$$\text{EMSE (dB)} = 10 \log \frac{P_1}{P_2} \quad (2)$$

where P_1 (watts) is received power with the fabric and P_2 (watts) is received power without the fabric. The input power used was 0 dB, corresponding to 1 W. The dynamic range (difference between the maximum and minimum signals measurable by the system) of the system was 80 dB [36]. The results of shielding effectiveness (SE) were evaluated according to ASTM D4935 standards. SE of the textile materials was measured in dB and percent values corresponding to these dB values were pinned down referring to FTTS-FA-003 [37].

3. RESULTS AND DISCUSSION

3.1. Morphology of Nanofibers

Solution property is highly influent about the adjustment of electrospun fiber diameter and morphology. Table 1 illustrates the viscosity and conductivity measurement results for individual electrospinning solutions of Pure PA6, PA6 with 1wt.% of AgNP and PA6 with 2 wt.%. of AgNP. When the amount of AgNP was increased more than 2wt.% in the blend, some limitations about forming stable jet during electrospinning was observed. This was most probably due to increased agglomeration among AgNP at higher concentration in the solution. Increment of AgNP in the solutions caused an increase in viscosity due to increase in solution concentration with the incorporation of AgNP. Conductivity also reached to 5.12 µS/cm for the PA6 with 2wt.% AgNP due to the increased amount of conductive metal particles in the solution.

It is commonly accepted that the solution viscosity is affected by polymer molecular weight and polymer/solvent blend ratio [38]. In our case, metal particles also contributed to the increase of solution viscosity. On the other hand, electrical conductivity of the solution is obviously critical for the generation of non-beaded nanofibers. Increase in the electrical conductivity of the solutions (charge volume density) can result in electrospun cylindrical fibers and finer fiber diameters since the polymer solution is subjected to more stretching under the high electrical field [39,40]. In Figure 3, SEM micrographs of electrospun nanofibrous membranes are presented. It was observed that beadless, mostly uniform and randomly distributed fibers were yielded. Also, very thin and highly porous webs among the fibers were formed which may be due to the uncontrolled drops of the solution from the nozzle (at top) to the rotating collector (on the ground) during electrospinning. Average fiber diameters were measured as 214±159 nm for Pure PA6, 174±70 nm for PA6 with 1 wt.% and 238±162 nm for PA6 with 2 wt.%. In addition, thicknesses of electrospun nanofibrous membranes ranged from 0.138 mm to 0.156 mm (Table 2). Furthermore, it was found that AgNP particles with small size along with agglomerated AgNP were dispersed whole over the membrane by either embedded on the fiber surface or located among the fibers (Figure 3).

Table 1. Solution properties and electrospun nanofibrous membranes characteristics

Polymer	Viscosity (cP)	Conductivity (µS/cm)	Nanofiber Diameter (nm)
Pure PA6	988	4.49	214±159
PA6 with 1 wt.% AgNP	1035	4.51	174±70
PA6 with 2 wt.% AgNP	1643	5.12	238±162

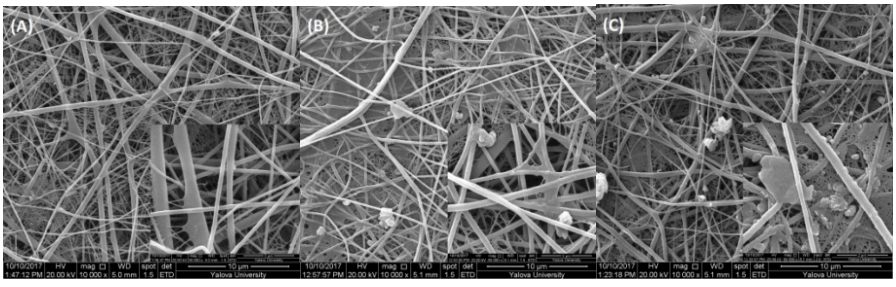

Figure 3. SEM pictures of electrospun nanofibrous membranes at 10000x and 50000x magnifications; (A) Pure PA6, (B) PA6 with 1wt.%, (C) PA6 with 2wt.%

It was noticed that much bigger and very thin sizes of fibers were fabricated together through the same system without changing processing parameters. This situation enlarged the fiber size distribution enormously for almost each nanofibrous membrane. Some justifications may be stated for this circumstance. Since our experiments were conducted in the vertically designed (from top to bottom) electrospinning set up, feeding rate of the solution to the electric field might be influenced from the feeding direction (opposite or in the same direction of gravity) and irregular polymer jet acceleration towards the collector.

Yarin et. al. reported that jet surface area increases significantly when the jet undergoes high stretching and elongation as a result of the electrical forces. Such increment in the jet surface area immediately accelerates solvent evaporation. Solvent rate decreases fast in the bending loops (approximately 90% solvent evaporates), only a few centimeters below the starting point of the bending (whipping) instability. Once the polymer concentration achieves to around 90%, the jet sustains elongating, but at a much lower rate. The decline in the elongation rate is because of the increment in viscosity and elastic modulus of the solution at greater polymer concentrations [41]. During our experiment, due to the irregular polymer jet acceleration –most probably because of the agglomerated particles in the solution-, the cross-sectional radius of the jet might be altered much while very thick and thin fibers were produced at the same time. However, the obtained fibers were still bead free.

Feeding direction may be another influent factor for the enlarged fiber distribution, because, in our previous experiments performed in another machine where the solution was fed from bottom to top, a fibrous structure with a more homogenous fiber size distribution was obtained even if the process conditions and solution concentrations were similar to the current study [14]. Therefore, feeding direction which was towards to gravity might cause the irregular feeding of the polymer solution into the electric field which induced production of very thin and thick fibers at the same time.

3.2. Mechanical properties of electrospun nanofibrous membranes

The tensile strength of a material expresses how much stress the material will tolerate before suffering permanent deformation/tearing. According to the tensile strength graph (Figure 4), the highest average values both in machine direction (11,13 MPa) and width direction (8,18 MPa) were recorded for Pure PA6 nanofibrous membrane. Tensile strength of the membranes declined gradually as the amount of AgNP increased in the membrane. This may be explained by the deformation of the membrane integrity to a certain degree, due to the locating of AgNP on the fibers surfaces and the pores among the fibers. On the other hand, the tensile strength of the nanofibrous membranes was found slightly greater in the machine direction (MD) comparing with the width direction (WD). However, difference between MD and WD was found greater for pure PA6 compare to other samples. The reason behind of this result may be the more parallel fibers laid along MD because of the rotating drum.

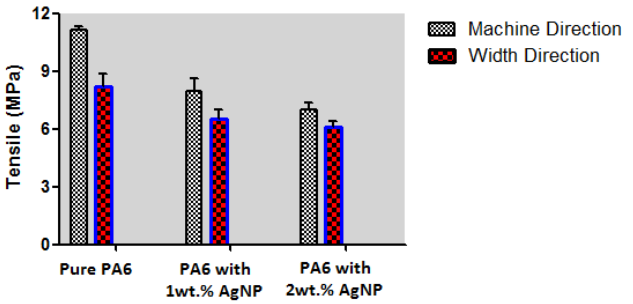

Figure 4. Tensile strength of electrospun nanofibrous membranes

In literature, there are several comments about the impacts of nanoparticles on the elongation properties of the electrospun membranes. For instance, An et. al. reported that the addition of nanoparticles to the polymers usually results in a reduction of the strain at break. On the other hand, Wang et. al. determined that the elongation at break increases by addition of nanoparticles [42,43]. In our case, elongation values decreased with introducing AgNP to the membranes. This may be attributed to the increase in stiffness of the membrane with the increase in AgNP concentration. Augmented concentration may lead to an increase in agglomeration, thus, higher stiffness and lower elongation [44]. Therefore, the highest average value was obtained for the Pure PA6 membrane in both machine (73%) and width (67%) directions.

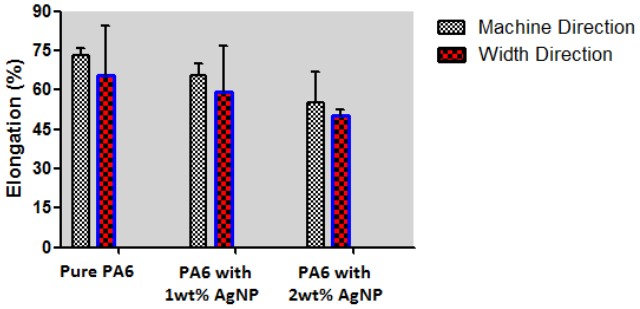

Figure 5. Elongation (%) of electrospun nanofibrous membranes

3.3. Electromagnetic shielding (EMSE) properties of electrospun nanofibrous membranes

In previous studies, several metal oxide nanofibers have been electrospun from sol-gel solutions and metallorganic precursors in order to obtain conductive structures [45,46]. Chen et. al. prepared conductive nanofibers by electrospinning of poly(methyl methacrylate) and silver trifluoroacetate in an organic cosolvent of methyl ethyl ketone and methanol. The final product gained from this study exhibited sheet resistances as low as 15 Ω/sq and total diffusive transmittances of 54% [47]. Demirsoy et. al prepared composite nanofibers of PAN with 1 wt.% and 3 wt.% $AgNO_3$ content by electrospinning. The conductivity of the nanofibrous membranes was measured as appproximately 10-8S/cm [44]. Erdem et. al. sputter coated the electrospun PA6 nanofibers with gold and palladium to yield functional nanofibrous membranes that possess electromagnetic shielding ability. The coated membranes exhibited almost 14 dB EMSE [14].

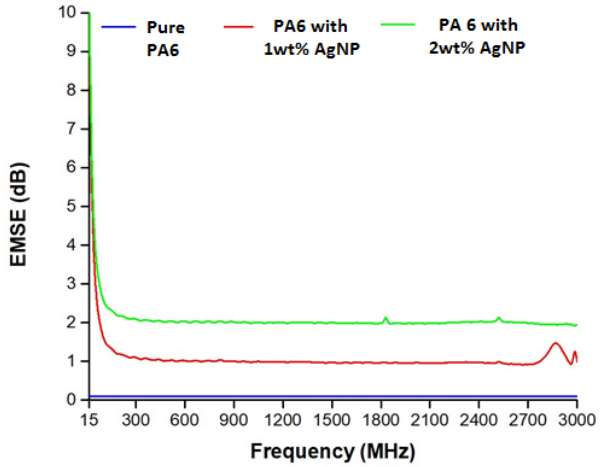

Figure 6. Electromagnetic shielding effectiveness (EMSE) of electrospun nanofibrous membranes

In our case, simple electrospinning process was performed to gain conductive membranes without the requirement of any further treatment. Figure 6 shows EMSE values of PA6/Ag nanofibrous membranes in 15-3000 MHz frequency ranges. The EMSE values of all PA6/Ag nanofibrous membranes showed a steady advance with the increment in frequency. Test results have clearly proved that EMSE is related to amount of Ag used in nanofibrous membrane because of increasing conductivity with AgNP fillers. The EMSE values of PA6/Ag nanofibrous membranes in the 15-3000 MHz frequency range were not high and below 3dB. Pure PA6 membrane did not perform any EMSE activity since it did not possess any conductive feature. AgNP have great potential to form highly conductive materials due to the increased surface area. Nanofibrous membranes are also very suitable to create a kind of networking environment for the interaction of conductive nanofillers to each other, regarding establishing flexible and efficient conductive platforms. To increase the EMSE performance of such materials, selection of proper polymers and nanofillers, and optimization of processing parameters are very crucial.

4. CONCLUSIONS

In this study, EMSE and mechanical properties of PA6 nanofibrous membrane with different Ag nanoparticles concentrations produced by electrospinning were investigated. PA6/Ag nanofibrous membrane in the 15-3000 MHz frequency range depicted better performance than those of pure PA6. The results show that in order to achieve higher shielding performance, the percentage of the nanofiller (Ag) in the nanofibrous membrane must be increased to an extent not to encounter challenges in electrospinning process. The highest electromagnetic shielding found in this work was around 2 dB for PA6 nanofibrous membrane with 2 wt.% Ag. More precise control of electrospinning process as well as fortification of the solution by incorporating auxiliary chemicals may impede or minimize AgNP precipitation which also yields achievement of nanofibers with better morphology. This well dispersion may positively affect fiber size distribution in the matrix which is also related to improved mechanical strength. Feeding process from top to bottom ensures the existence of AgNPs in the fibers and should be well controlled to prevent dripping. Consequently, more precise control of these processes leads to either incorporation of more AgNPs in the solution (better EMSE values) or improved mechanical properties.

Acknowledgement

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(REFEREED RESEARCH)

YAPAY GÖRME TABANLI KUMAŞ HATA TESPİT SİSTEMİ

ARTIFICIAL VISION BASED FABRIC DEFECT DETECTION SET-UP

Devrim Demiray SOYASLAN[1], İbrahim KARATAŞ[2]

[1]Polymer Eng. Department, University of Mehmet Akif Ersoy, Burdur, Turkey
[2]Dep. of Material Technologies Eng., Institute of Science and Technology, Burdur, Turkey

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ABSTRACT

Due to the cost and complexity of existing defect detection systems, a fabric defect detection device based on an artificial vision system has been developed in this study. Using knitted pile fabric, six types of defects were studied: loop drop, fly defect, grease spot, cross- striped defect, hole defect and pilling defect. Obtained fabric images were converted into histograms by a computer program developed within the scope of this study and defect types were characterized.

Keywords: Artificial vision, defect detection, textile, fabric

ÖZET

Mevcut hata tespit sistemlerinin pahalı ve komplike olmalarından yola çıkılarak bu çalışmada, yapay görme sistemi tabanlı bir kumaş hata tespit cihazı geliştirilmiştir. Örme havlı kumaş kullanılarak ilmek düşmesi, uçuntu hatası, yağ lekesi, enine yönde çizgi hatası, patlak ve boncuklanma olmak üzere altı tip hata üzerinde çalışılmıştır. Elde edilen kumaş görüntüleri yine bu çalışma kapsamında geliştirilen bir bilgisayar programı ile histogramlara dönüştürülmüş ve hata tipleri karakterize edilmiştir.

Anahtar Kelimeler: Yapay görme, hata tespit, tekstil, kumaş

Corresponding Author: Devrim Demiray Soyaslan, dsoyaslan@mehmetakif.edu.tr

1. INTRODUCTION

Defect detection in textile fabrics are still made mostly by human inspectors using a fabric control machine. The fabric on the control machine, moves through the inspectors' visual field and the inspector have to detect these small defects. In general, the identification rate is 70% for controls performed with this method and this ratio decreases with fatigue (1). So, human inspectors fail on detection defects in terms of accuracy, consistency and efficiency (2, 3). The first industrial studies related to computer vision are based on 70s (4). At that time, the cost of computing power was very high, and this was an obstacle in the development of image analysis techniques. But with the recent advances in computer and imaging technology, low-cost software and programs have emerged and these have enabled effective image analysis techniques to evolve. This has made automated image analysis detection systems an attractive alternative to human inspection (5, 6).

The first considerations in textile about image analysis were textile quality control, evaluation of trash content in cotton and evaluation of blend irregularity in blend yarns (7-9). Studies about detecting and classifying fabric defects with image analysis have been began to carry out in 1980s and these studies were firstly on woven fabrics (4, 10-19). A few research about detecting the fabric defects has been studied on knitted fabrics (20-23).

Existing defect detection methods are collected in three categories: statistical, spectral and model-based. Each category has its own specific use area (3, 24). Example, statistical methods are not useful for the detection of textured defects, such as woven and knitted fabrics. Therefore, spectral methods such as Fourier transform (25-27) are used to detect these kind of fabric defects. Nearby another approaches using wavelet or Gabor functions (28-32) have also been used for fabric detections.

All these literature have given important datas about defect detection systems but none of them was studied on the relation between the defects and histograms. However the histograms are very clear and easy graphics for inspectors

to understand and notice the defect while the fabric is flowing through the 'fabric control machine'.

In this study, knitted fabric defects are tried to define with histograms using a very simple and cheap artificial vision based fabric defect detection set up.

2. MATERIAL AND METHOD

2.1. Material

In the study, the knit pile fabric made from 100% cotton that has7 loop/cm course density, 12 loop/cm wale density and 150 gram/m^2 fabric weight was used.

Arduino nano circuit board was used to transfer the data obtained from fabric by way of TSL201R optical line sensor to Java software, to process it with the help of Java programming language, and then to compose histograms. TSL201R optical line sensor is exerted with the purpose to acquire data over fabric. The charge accumulation amount of each pixel is directly proportional to the light density and the integration time. The gap between two sequential outputs is identified as integration pitch.

2.2. Method

The user interface is made up as a desktop application in C# programming language at the Microsoft Visual Studio 2015 platform. Within the program, there is an integrated java library. Histogram graphics were edited by way of the software encoded in java programming language at processing 3.0.1 program. Operating Arduino nano and TSL201R optical line sensor, the defect detection device designed and advanced in this study.

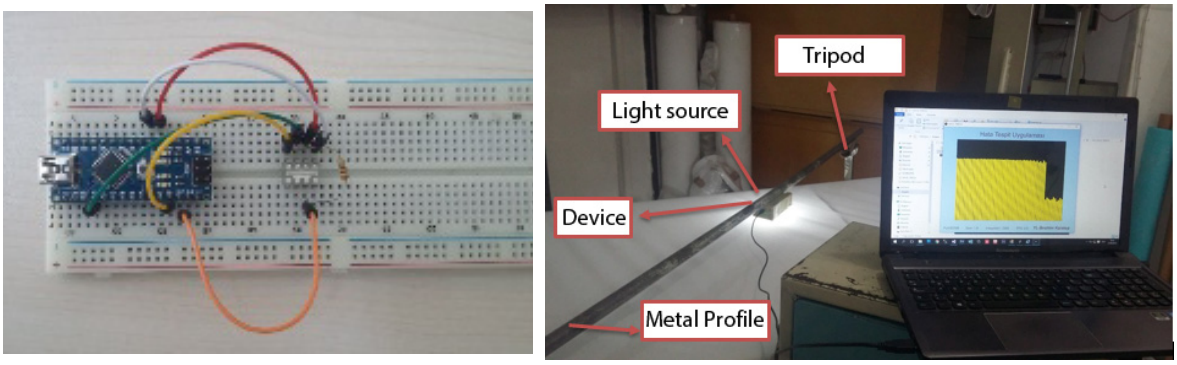
A single optical line sensor can scan a length of 11.55 cm. The data interval increases as much as the usage angle of optical line sensor scales up. Yet, as moving away from the surface of which its image would be taken, the measurement would not be carried out in the expected quality and accuracy since the data inspected by optical sensor would be affected negatively by environmental factors such as light. For this reason, in this study, the defect detection device was fixed at optimum 10 cm height which is proposed by TSL201R optical line sensor producer TAOS firm and then measurement was done.

As observed on Figure 1, the defect detection device transformed into a closed box was mounted over a metal profile with fixed stays to enhance the accuracy of optical sensor and to shorten the integration time. Thereby, the defect detection device could be isolated from external effects such as noise and light. The device was connected with a laptop computer via 5V usb cable with intent to process the detected data. The fabric flow was enabled between two cloth take up machines which were installed in opposing sides. White fluorescent light source with 302.5 mm length was placed just below the defect detection device in the midst of two cloth take up machines. In this study six types of defect, namely loop drop, fly defect, grease spot, cross-striped, hole, piling were sought to uncover. The 30 meter roll of knit pile fabric was transferred wrapping from one machine to another between two cloth take up machines with a speed of 17.7 meter per minute. The defect detection is performed with only one sensor so the sensor has scan only the length of 11.55 cm on the fabric.

The fabric was passed three times through the same scanning. The histogram graphs of each defect type obtained from the three scan results were compared and it was discovered that the three histogram graphics of each defect type were similar to each other. The number of frame per second taken by the defect detection device is accounted as 10.6 fps in the best achieved integration time (536 milliseconds). In the similar studies using the artificial vision system, the fps values were measured in range of 2 and 25 fps (33-35).

According to the autocorrelation based approach which is among the statistical approaches, the defect detection can be procured examining regular structures. In case any data is discerned out of the regular structures, the riffles and fluctuations come into existence on histograms.

The system's operation principle relies on capturing a wavelength over the light reflected on sensor from the light source located at the back of fabric. This wavelength remains stable throughout the fabric surface. Yet, as the sensor faces any defect, the light amount reflected on sensor shifts hence, the riffles and fluctuations occur on histograms. The flow diagram of the procedure is drawn and showed in Figure 2. In the diagram the WL abbreviation describes the wavelength of the light.



The integration of Arduino Nano and TSL201R optical line sensor on board

Figure 1. Application of Defect Detection Device

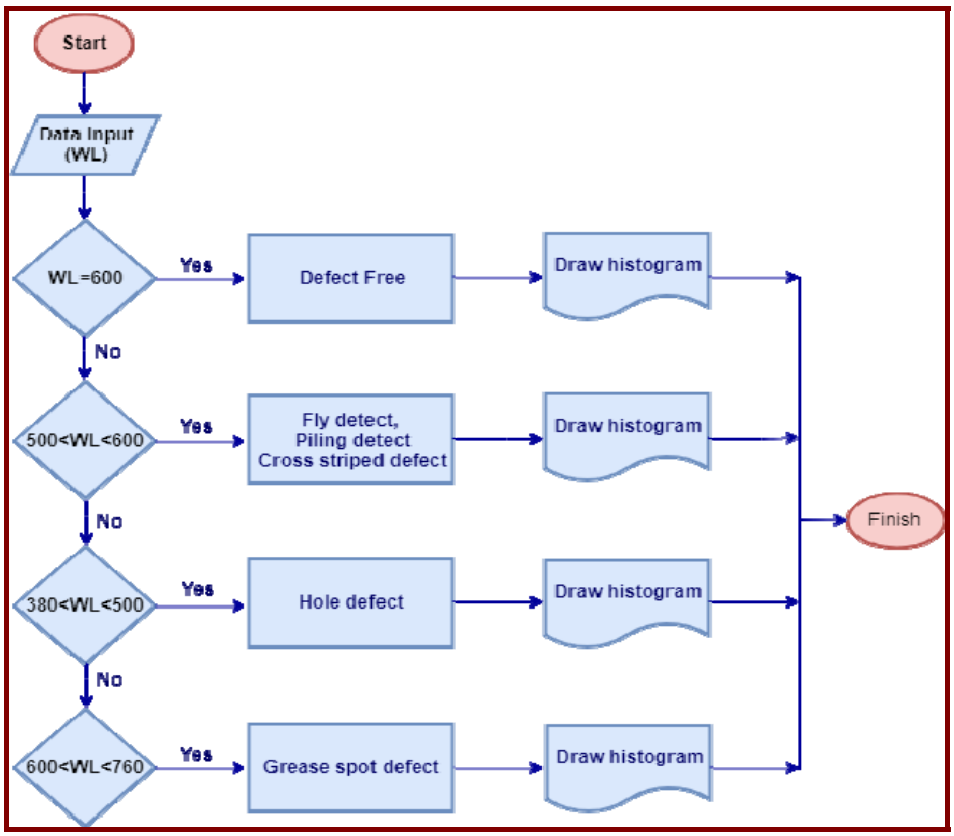


Figure 2. The flow diagram of the procedure

3. RESULTS AND DISCUSSION

The data obtained from the advanced defect detection device was evaluated by the autocorrelation based approach and the results explained in detail below were gotten. As aforementioned in previous sections, the reason to choose the autocorrelation approach is the quite conserved structure of autocorrelation method against enlightening and noise variations and this structure's superiority over other statistical approaches. In addition, the classification accuracy rate of autocorrelation method on certain fabric types is higher than those of morphological approaches.

In this study, the prototype of defect detection device at minimum cost was advanced operating Arduino Nano and TSL201R optical line sensor and the defect detection was carried out basing upon the autocorrelation approach. Owing to the autocorrelation method, the images taken from the defect detection device were transformed into histograms in accordance with defect types (riffle, valley, concave).

The image of defect free fabric and its histogram are shown at Figure 3. As can be understood from the figure; the histogram of the defect free fabric gave a perfect graph. Any riffle or concave form were not occured in the graph because the amount of light passing through all the surface of the fabric is the same as there is no faulty region in the fabric. So defect free fabric histogram is exactly different from the other defective fabric histograms.

The image of hole defect on fabric and its histogram are indicated at Figure 4. As shown in the figure, the hole zone emerges in riffle form due to the maximum light penetration at this yard.

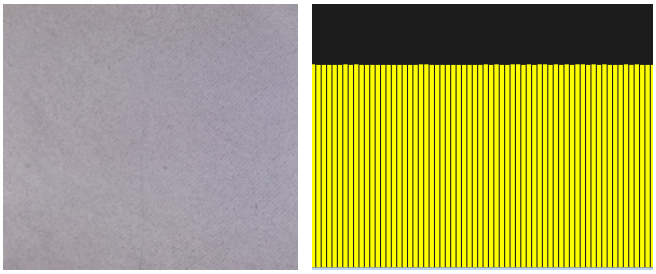


Figure 3. The image of defect free fabric and its histogram

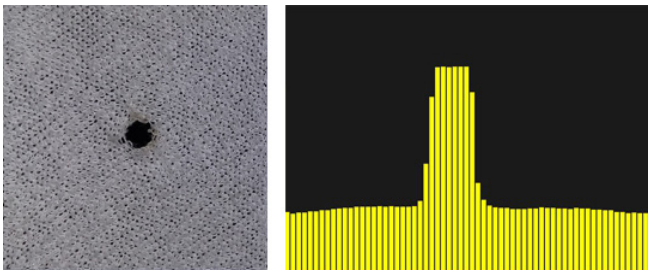


Figure 4. The image of hole defect on fabric and its histogram

The image of grease spot defect on fabric and its histogram are shown at Figure 5. The grease spot obturates fabric pores and to prevent majorly the light to traverse as it can be assumed from the fabric image. Therefore the fabric area where the grease spot is intense creates a concave shape on histogram since this zone is impenetrable to light.

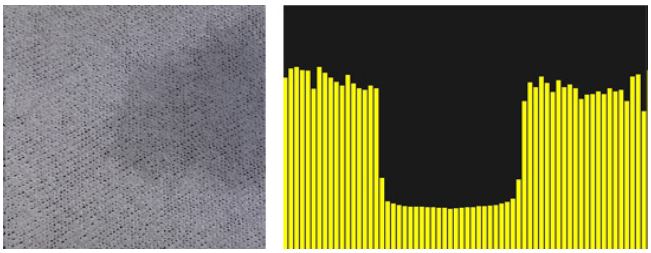


Figure 5. The image of grease spot defect on fabric and its histogram

The image of loop drop defect on fabric and this defect's histogram is shown at Figure 6. Comparing to the histogram of defect free fabric, the emergence of slight riffle is observed. It is deemed that this riffle forms as the feet, leg and head parts of sporadically dropping loops fill the gap of fabric.

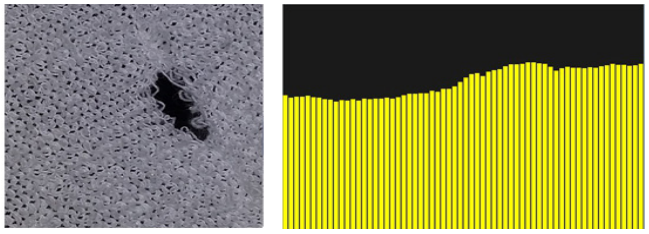


Figure 6. The image of loop drop defect on fabric and its histogram

The image of fly defect on fabric and the histogram resulting from this defect are brought forth at Figure 7. The concaves are composed intermittently whereabouts fly defects appear because light amount reaching optical sensor is lower there.

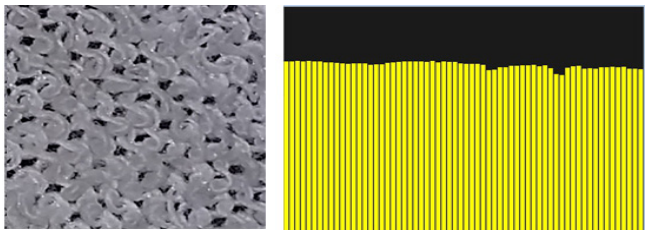


Figure 7. The image of fly defect on fabric and its histogram

The image of cross-striped defect on fabric and its histogram are displayed at Figure 8. This defect's histogram looks like that of fly defect (Figure 7) as it is apparent on the figure. This is why the white light under the fabric attained the optical sensor in some areas while reaching the sensor at a low level or none at all in other areas at the cross-striped defect.

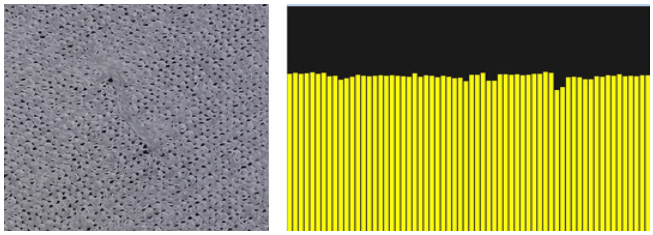


Figure 8. The image of cross-striped defect on fabric and its histogram

The image of pilling defect on fabric and its histogram are shown in Figure 9. The pilling defect is similar to the fly defect in terms of apparition on fabric. The fibers get lumpy at pilling defect and these lumps remain tied to the fabric. During the process of defect detection, the white light transmits less light where these lumps locate and it composes the concave at histogram.

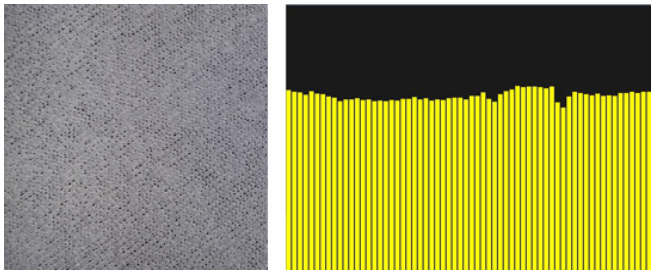


Figure 9. The image of pilling defect on fabric and its histogram

Within the extent of study, the defect detection device advanced to identify certain defect types on knit pile fabric carried out requested measurements at the expected accuracy value. In consequence of the literature review, it was observed that the histograms acquired from the defect detection device developed within this study are similar to those in the related studies (9, 19, 33-35).

4. CONCLUSION

The cost of artificial vision systems in industrial domains is quite high. The installation and disposal of these systems require high amount of budget for producers. The human-oriented defect detection systems are still in use to identify defects at small enterprises. This study goes into the design and active operation of an artificial vision-based defect detection device alternative to existent expensive devices thanks to its low-budget. Six types of defects on knit pile fabric, namely fly defect, loop drop, grease spot, hole, pilling, and cross-striped defect were examined using the artificial vision based defect detection device which was advanced within the scope of this study. The fly defect, pilling, and cross-striped defect form alike histogram graphics (creating in places riffles and valleys). Its reason is assumed as that pilling and fly defects generate similar views on fabric, yet almost identical histogram graphic of cross-striped defect to those of pilling and fly defect is evaluated as an unexpected situation.

It is found that the defect detection device has decent accuracy to fly, pilling, and cross-striped defects, however it has not the expected discernibility capacity. Based on this fact, it is deduced that the discovery of fly, pilling, and cross-striped defects needs the micro-scale examination.

In the future, for the second part of this study; defect detection will be performed with multiple sensors to scan the entire width of the fabric. In addition, statistical analyzes of datas will be added to the study.

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(REFEREED RESEARCH)

HVI LİF ÖZELLİKLERİNİ KULLANARAK KOMPAKT İPLİKLERİN MUKAVEMET VE DÜZGÜNSÜZLÜKLERİNİN TAHMİNLENMESİ

ESTIMATION OF TENSILE STRENGTH AND UNEVENNESS OF COMPACT-SPUN YARNS BY USING HVI FIBER PROPERTIES

Memik Bünyamin ÜZÜMCÜ[1*], Hüseyin KADOĞLU[2]

[1]Bartın University, Department of Textile Engineering, Bartin, Turkey
[2]Ege University, Department of Textile Engineering, Bornova / İzmir, Turkey

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ABSTRACT

Compact spinning system, which has begun to gain an important place in textile industry, thanks to its desired yarn characteristics, was developed in the early 2000s. Less hairy yarns can especially be produced with this system, as stated in the literature. Moreover, the system has significant advantages in terms of tensile strength. In this study, yarns were produced with different yarn counts (Ne 40 - Ne 90) and different twist coefficients (αe 3.8, αe 4.2 and αe 4.6) using the compact spinning system. Regression analysis was carried out using the test results of the yarns produced and HVI fiber properties which were measured before the production. It was investigated whether the properties of the yarns can be estimated before production by using specified fiber properties. As a result of the analysis, the derived equations were used to estimate tensile strength and unevenness values of the yarns.

Keywords: Compact spinning system, fiber properties, HVI, yarn properties, estimation.

ÖZET

Tekstil endüstrisinde, sunmuş olduğu farklı iplik karakteri sayesinde, önemli bir yer edinmeye başlayan kompakt iplik eğirme sistemi 2000'li yılların başında geliştirilmiştir. Bu sistem ile, literatürde de belirtildiği üzere, özellikle daha az tüylü iplikler üretilebilmektedir. Bunun yanı sıra, mukavemet açısından da sistemin önemli avantajları bulunmaktadır. Bu çalışmada kompakt iplik eğirme sistemi kullanılarak farklı numaralarda (Ne 40- Ne 90 aralığında) ve farklı büküm katsayılarında (αe 3.8, αe 4.2 ve αe 4.6) iplikler üretilmiştir. Üretimler öncesinde ölçülen HVI lif özellikleri ile üretilen ipliklerin test sonuçları kullanılarak regresyon analizi yapılmıştır Analiz sonucunda lif özellikleri belirlenmiş olan ipliklerin, özelliklerinin üretim öncesinde tahminlemelerinin yapılıp yapılamayacağı incelenmiştir. Analiz sonucunda türetilen denklemler ile mukavemet ve düzgünsüzlük değerlerinin tahminlemesi yapılmıştır.

Anahtar Kelimeler: Kompakt eğirme sistemi, lif özellikleri, HVI, iplik özellikleri, tahminleme

Corresponding Author: Memik Bünyamin Üzümcü, muzumcu@bartin.edu.tr

1. INTRODUCTION

Compact spinning system was developed at the beginning of the 2000s and has become a preferred system in the market, due to characteristics of the yarns it produces. This system enables the production of yarns with lower hairiness and higher tensile strength, as described in the literature (1-4). As is known, major reason of hairiness is spinning triangle which occurs at the exit of the delivery cylinders (5).

Compact spinning system was developed with modifications made to the drafting zone of ring spinning system and the aim of this was to eliminate the spinning triangle. Although it is not completely possible to eliminate the spinning triangle, the base length of the spinning triangle can be reduced by compacting the fiber bundle with the parts added to the drafting zone. Thus, the fibers which increase the hairiness by joining the yarn at one end are totally joined to the yarn body and that way the tensile strength can also be increased.

There are different types of compact spinning systems in the market. While most of them perform compacting using negative air pressure, some systems uses magnetic mechanical principles (6). In literature, there are studies comparing systems using negative air pressure, as well as comparing them with systems using magnetic mechanical principles (7-9). However, as a general result, most of the studies concluded that all of these compact spinning systems are superior to conventional ring spinning system, especially in terms of tensile strength and hairiness.

Fiber properties need to be known, as yarn producer have to adjust some machine parameters according to them

besides quality of raw material. According to spinning system, some fiber properties become more important in terms of spinning quality. Fiber length is crucial in ring and airjet spinning while fiber strength has more importance in open-end spinning system (10). HVI and AFIS systems are devices developed by Uster, which are used for determination of fiber properties. In fiber standardization, the HVI device has a great importance. Before this device; fiber properties such as strength, elongation and fineness were measured with Pressley tester, Stelometer and Fibronaire (11). After the development of the device, it became possible to obtain more detailed information about fiber in one device and to standardize it accordingly. With current technology, the device can test properties such as fiber fineness, length, strength, elongation, lightness, yellowness, trash and nep number (12). Some of these features were also used in our study.

Producers make a number of production experiments while developing new products or improving existing products. These experiments cause additional raw material, energy and labor costs. Estimation takes place at this point. By using the features of the inputs used in production, it is possible to estimate the results with statistical methods. There are studies in the literature where these methods are used for the textile industry. Some of these studies focus on estimation of yarn strength, breaking extension, unevenness, hairiness and IPI faults as well as fabric properties. Üreyen and Kadoglu used regression analysis for estimation of ring spun yarns' tensile strength, breaking elongation, unevenness and hairiness values by using HVI and AFIS fiber properties (13,14). Faulkner et al also investigated the effects of fiber properties (used HVI and AFIS test results) to ring-spun yarn properties by using regression analysis (15). Üreyen and Gürkan used regression analysis and artificial neural network, in order to predict hairiness and unevenness values of ring-spun yarns (16). Furferi and Gelli used artificial neural network for predicting tensile strength of ring-spun yarns produced from different fibers by using fiber properties (17). Bedez Üte and Kadoglu used regression analysis to estimate tensile strength and unevenness of siro-spun yarns by using HVI and AFIS properties of the fibers used in productions (18,19). Uzumcu and Kadoglu used HVI fiber values for predicting IPI fault values of compact-spun yarns (20). Gurkan Unal used artificial neural network and response surface methods for evaluating the effects fiber properties on spliced yarn characteristics (21,22). For predicting the pilling tendencies of cotton interlock knitted fabrics, Kayseri used both regression analysis and artificial neural network methods in their studies (23,24). Sari and Oglakcioglu also used regression analysis in their study about pressure characteristics of medical stockings (25). Not only for cotton yarns, cotton waste containing yarn properties were also tried to be explained by using correlation with fiber properties by Telli and Babaarslan (26). Positive results were obtained in most of these studies where various methods such as regression analysis and artificial neural networks were used for estimation.

In this study, it was investigated that how fiber properties effect yarn properties and if it is possible to determine yarn properties before production, in case of using compact spinning system. For this purpose, yarns were produced by compact spinning system using rovings produced from different blends. Data gathered from previous studies about predicting yarn properties indicated that various types of analysis were performed for properties of coarse (≤Ne 40) yarns produced with ring, rotor, siro and vortex spinning systems. In our study, fine yarns (≥Ne 40) were produced by using compact spinning system and regression analysis was performed by using the properties of the fibers measured by HVI and some yarn parameters (yarn count, twist coefficient) and as a result the estimation equations were developed.

2. MATERIAL AND METHODS

In this study, it was aimed to estimate the properties of cotton compact-spun yarns. For this reason, cotton fibers of different properties were used and initially properties of these fibers were tested. Uster HVI was utilized in determining the fiber properties. Fiber tests were carried out after combing process, because resultant fiber properties were needed for our estimation equations.

Rovings from different cotton blends were produced in Söktaş Dokuma İşletmeleri San. Ve Tic. A.Ş. Rovings were produced with two different counts (Ne). These roving counts were Ne 1.3 (for 7 blends) and Ne 2.0 (for 4 blends). Thus, yarn productions were started by using 11 different blend types. Unevenness values of the rovings used in the productions were measured with Uster Tester 5 device.

Machine settings which should be used in the production were determined before the yarn productions started. For this purpose, we interviewed industrialists and moreover, made a preliminary experiment using Taguchi experimental design, which included the factors of spacer type, spindle speed and ISO of traveller. In this preliminary experiment, three different types of spacers, three weights of traveller and three speed for spindle were used in the range suggested by the machine manufacturer, and the levels that provided the highest strength and the lowest unevenness / hairiness values according to the Taguchi method were preferred in the productions. By the means of yarn count, yarns were produced starting from the thinnest yarn number that can be produced with the rovings in hand, to thicker numbers. Productions of six different yarn counts (Ne 40, Ne 50, Ne 60, Ne 70, Ne 80 and Ne 90) were intended, after preliminary tests for both roving counts (Ne 1.3 and Ne 2.0). However, due to technical limitations of the machine, yarns finer than Ne 70 could not be produced by using rovings with Ne 1.3 count. The final production plan created as a result of the preliminary studies is given in Table 1 and the machine settings used in the productions are given in Table 2. Productions were carried out using Rieter K45 compact spinning machine.

One of the most important things taken into consideration in yarn production was that productions were always done using the same spindles. For this reason, firstly, a code number was given to rovings which were tested according to the standard. In the second stage, 20 spindles of which had non-problematic drafting and twisting zones were selected and given code numbers. These code numbers of roving and spindles were written on each of the produced cops. Therefore, the results obtained from the tests were evaluated considering which roving and which spindle was used in the production of the yarn. As a precaution against errors that may occur in tests, backup for every single cop was produced.

Table 1 Production plan

Roving count (Ne)	Yarn count (Ne)	Twist coeeficient (α_e)		
		3.8	4.3	4.8
1.3	40	+	+	+
	50	+	+	+
	60	+	+	+
	70	+	+	
2.0	40	+	+	+
	50	+	+	+
	60	+	+	+
	70	+	+	+
	80	+	+	+
	90	+	+	

Table 2 Machine settings

Yarn count (Ne)	Spindle speed (rpm)	Spacer (mm)	Traveller (ISO)
40	17000	3.25	31.5
50		3.25	25
60		3.00	22,4
70		3.00	20
80		2.75	18
90		2.75	16

For each yarn type, ten cops were used in the tests. These cops were conditioned and subjected to yarns tests according to the standards. Unevenness tests were performed using Uster Tester 5 and Uster Tensorapid 4 was used for tensile strength tests.

Data gathered from the tests were used for correlation analysis by using SPSS and for regression analysis using GRETL. Correlations between the fiber properties were determined and fiber properties with least correlation with other fiber properties were selected in order to be used in further analysis. In the regression analysis part, control tests were performed after analysis. With these tests, heteroscedasticity, collinearity and distribution of residuals were examined. The validity and reliability of the estimation equations were ensured by making necessary corrections according to results of these tests.

3. RESULTS

In this study, blends with different mean fiber properties were used and the effects of fiber properties on yarn properties were investigated. Initially, fiber properties were obtained by using Uster HVI. Test results are given in Table 3.

In accordance with the aim of the study, ANOVA test was conducted to determine whether the fiber test results of blends were statistically different. Results of this test showed that differences between blends were statistically significant (p=0.00 <0,05).

Correlations between fiber properties and yarn properties (yarn count and twist coefficient) which were planned to be used as independent variables were also examined. The results of this analysis are given in Table 4. According to the correlation in between fiber properties (if they were high), some of them were not used in regression analysis, as mentioned before. The reason for this was that the use of two highly correlated fiber values together in the regression analysis would have caused a virtual rise in the relationship between fiber properties and yarn property which was aimed to be estimated. Then, correlation between the fiber properties and the measured yarn properties was investigated. What is important here is that generally, fiber properties which had a relatively high correlation with the yarn property that would be estimated were used. The results of the analysis are shown in Table 5.

Table 3 HVI results of 11 different blends

Blend	UHML	UI	Str	Elg	SFI	Mst	Mat	Mic	Rd	+b	SCI
B1	30.57	83.35	39.50	5.3	5.95	5.70	0.870	4.105	79.65	10.10	171
B2	31.57	84.50	43.40	5.3	5.60	5.90	0.885	4.840	78.15	11.70	182.5
B3	31.53	84.10	39.10	5.5	5.70	5.80	0.880	4.650	77.10	11.70	169
B4	31.19	84.00	40.80	4.9	5.75	5.55	0.880	4.610	75.05	11.25	172
B5	30.28	84.30	41.00	5.2	5.90	5.25	0.890	4.965	82.80	9.45	173
B6	26.42	83.30	28.30	4.7	7.50	6.00	0.895	5.230	80.30	11.35	121
B7	26.87	83.20	29.85	4.6	7.35	5.65	0.890	4.895	79.75	11.05	129
B8	31.67	83.95	39.80	5.4	5.90	6.00	0.880	4.750	77.90	11.35	171
B9	30.38	82.30	40.10	5.1	6.50	5,50	0.890	4.900	73.30	12.10	158
B10	30.36	83.75	39.65	4.9	5.85	5.50	0.880	4.585	80.35	10.85	170
B11	27.70	84.05	30.60	5.2	6.40	5.65	0.880	4.810	80.65	10.85	138

Table 4 Correlation analysis results for HVI fiber properties

	(Ne)	(T/m)	SCI	MIC	STR	LEN	UNF	SFI	ELG	Rd	B	Mat
(Ne)	1	0.751*	0.148*	-0.101*	0.177*	0.173*	-0.077*	-0.097*	0.153*	-0.174*	0.106*	-0.069*
(T/m)	0.751*	1	0.142*	-0.090*	0.167*	0.162*	-0.056	-0.095*	0.131*	-0.145*	0.079*	-0.061
SCI	0.148*	0.142*	1	-0.492*	0.969*	0.955*	0.437*	-0.950*	0.693*	-0.179*	-0.086*	-0.454*
MIC	-0.101*	-0.090*	-0.492*	1	-0.375*	-0.443*	-0.028	0.519*	-0.393*	-0.004	0.371*	0.931*
STR	0.177*	0.167*	0.969*	-0.375*	1	0.956*	0.262*	-0.868*	0.630*	-0.348*	0.049	-0.314*
LEN	0.173*	0.162*	0.955*	-0.443*	0.956*	1	0.321*	-0.908*	0.744*	-0.390*	0.126*	-0.447*
UNF	-0.077*	-0.056	0.437*	-0.028	0.262*	0.321*	1	-0.581*	0.404*	0.490*	-0.273*	-0.142*
SFI	-0.097*	-0.095*	-0.950*	0.519*	-0.868*	-0.908*	-0.581*	1	-0.755*	0.096*	0.112*	0.561*
ELG	0.153*	0.131*	0.693*	-0.393*	0.630*	0.744*	0.404*	-0.755*	1	-0.091*	-0.040	-0.494*
Rd	-0.174*	-0.145*	-0.179*	-0.004	-0.348*	-0.390*	0.490*	0.096*	-0.091*	1	-0.780*	-0.036
B	0.106*	0.079*	-0.086*	0.371*	0.049	0.126*	-0.273*	0.112*	-0.040	-0.780*	1	0.364*
Mat	-0.069*	-0.061	-0.454*	0.931*	-0.314*	-0.447*	-0.142*	0.561*	-0.494*	-0.036	0.364*	1

*Significant for p=0,01

Table 5 Correlation analysis results for yarn and HVI fiber properties

	Tensile Strength (cN/tex)	Breaking Elongation (%)	Unevenness	Thin Places (-50%)	Thick Places (+50%)	Neps (+200%)	Hairiness (H)
Measured Ne	0.003	-0.672*	0.652*	0.583*	0.438*	0.468*	-0.723*
Measured T/m	0.074*	-0.265*	0.477*	0.369*	0.305*	0.304*	-0.795*
MIC	-0.358*	-0.064*	0.179*	0.165*	0.286*	0.225*	0.084*
STR	0.851*	-0.029	-0.420*	-0.348*	-0.549*	-0.508*	-0.277*
LEN	0.827*	-0.038	-0.416*	-0.348*	-0.564*	-0.538*	-0.287*
UNF	0.292*	0.026	-0.382*	-0.198*	-0.326*	-0.302*	-0.051
SFI	-0.772*	-0.059	0.531*	0.404*	0.641*	0.595*	0.256*
ELG	0.581*	-0.091*	-0.276*	-0.239*	-0.486*	-0.501*	-0.260*
Rd	-0.165*	0.084*	-0.112*	0.037	0.037	0.068	0.197*
B	-0.152*	-0.146*	0.235*	0.087*	0.151*	0.119*	-0.069*

*Significant for p=0.01

Ordinary least squares method was used in regression analysis. The results of this multiple regression analysis technique, in which the effects of many independent variables to a single dependent variable can be examined, were obtained [27]. As a result of the analysis made for tensile strength, only statistically significant value was found to be the fiber strength. Tests were also carried out to determine the validity of the regression. The "White's test" was used to evaluate heteroscedasticity and because of negative result of this test, analysis was repeated with heteroscedasticity corrected option of GRETL. R^2 value was found to be 0.82 and Akaike criterion was 4896.96 (Table 6). The graph of measured vs estimated yarn strengths is given in Figure 1.

In the analysis for unevenness, the relation between the fiber properties and Uster % CV values of yarns was investigated, initially. For this purpose, curve fitting was used in the study, like Ureyen performed in his study and fiber properties, which were not in a linear relationship with the dependent variable, were converted into new variables to provide linearity (28). Fiber strength, micronaire and short fiber index, which had statistically significant role according to the analysis, were used in estimation equation. In order to reach the highest R^2 values, uniformity was excluded.

Foretold conversions were done and new independent variables were defined as follows:

- Quadratic fiber strength (QSTR)= 34.35 – 1.031 x fiber strength (STR) + 0.01305 x fiber strength^2 (STR^2)
- Cubic short fiber index (CSFI)= 286.3 – 130.6 x short fiber index (SFI) + 20.62 x short fiber index^2 (SFI^2) – 1.070 x Short fiber index^3 (SFI^3)

Regression analysis was performed using new independent variables obtained with conversions. Estimation equation was gathered by regression analysis and this equation and R^2 values are given in Table 7. The graph of measured and estimated unevenness values is given in Figure 2.

Table 6 Estimation equation for tensile strength

Estimated Value	Estimation equation	R^2	Adj. R^2
Tensile Strength (cN/tex)	1.57153 – 0.0342010 x Ne + 0.123803 x α_e + 0.149382 x CVm + 0.542162 x STR	0.82	0.82

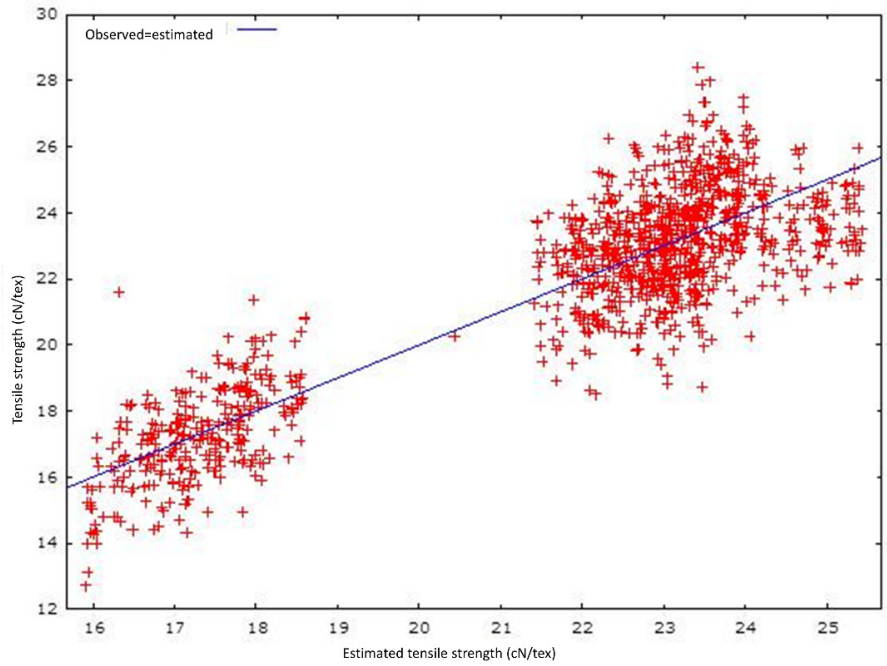

Figure 1 Estimated-Observed Tensile strength values

Table 7 Estimation equation for unevenness

Estimated Value	Estimation equation	R^2	Adj. R^2
Unevenness (cN/tex)	-6.29767+ 0.0583889 x Ne - 0.594462 x Mic + 0.815532 x CSFI + 0.598781 x QSTR	0.82	0.82

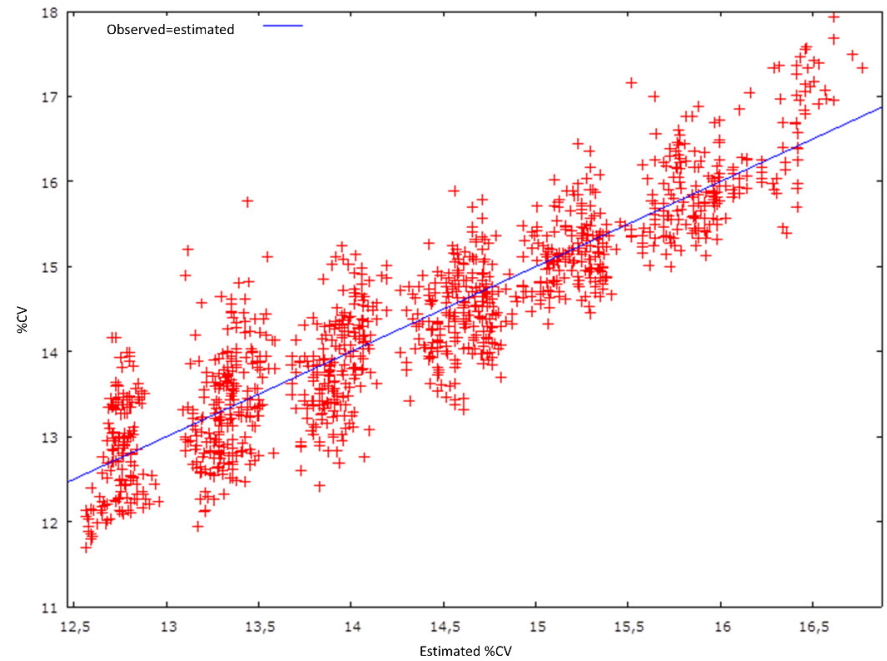

Figure 2 Estimated-Observed unevenness values

Both tensile strength and unevenness analysis were examined via controlling variance inflation factors (VIF). Collinearity was controlled by this way and in both analyzes, results of this test were found to be within the necessary limits.

4. DISCUSSION

The aim of this work was to determine the yarn properties by using raw material properties, before spinning. The estimation equations obtained for this purpose as result of the regression analysis were given. The data obtained in

these analyzes (approximately R^2 = 0.82 for both) were found at a satisfactory level.

When the studies of Bedez Üte on estimation of siro-spun yarn properties and Ureyen's on estimation of ring-spun yarn properties were examined, it was seen that both studies can determine the yarn strength and unevenness values well, by using the estimation equations developed by regression analysis (12,28). However, when these two studies were compared, it was found that regression analyzes for the ring-spun yarns resulted with higher R^2 values. Moreover, this value (R^2) seems to be even lower, in comparison with both studies, in our study. Though more investigation will be made in this matter, our first thought was that the siro spinning system, compared to the ring spinning system, brought yarn properties closer to the theoretically expected values by minimizing the problems caused by the system. Since this effect is more valid in the compact spinning system, our R^2 value was thought to be lower than the siro system. In other words, it was believed that the effect of the spinning system on the yarn is higher in these cases. In Siro spinning system, the two pre-yarns are twisted together and form a yarn, so that a problem that may occur in each yarn caused by any fiber property may be eliminated randomly. Likewise, in the compact spinning system, due to compacting, fibers that normally would not be able to join might be added to the yarn body and caused changes in yarn properties. That is to say, although the fiber properties were different from each other, system reduced effects of some of them which may normally have a negative effect on yarn properties. This topic is planned to be examined in detail in following studies.

In this study, it was also aimed to estimate hairiness, one of the most important yarn properties. However, as mentioned before, the correlation between fiber properties and hairiness was very low due to the effect of the system. Correlations of hairiness with fiber length and short fiber ratio which were normally expected to be high, were at the Pearson coefficient levels of 0.287 and 0.256, respectively, as can be seen from Table 3. For this reason, the estimation equation for hairiness value could not be developed with HVI data.

5. CONCLUSION

With this work, it was aimed to develop estimation equations which we hope to lead the industrialists who produce yarn and piled a lot of fiber and yarn data. It is believed that having prior knowledge of the yarn without increasing costs of additional raw materials, labor and energy may increase the competitiveness of the yarn producers. Positive results were obtained for tensile strength and unevenness (R^2 = 0.82). Yarns were produced to examine the estimation precision and their test results and the results obtained from the estimation equations were compared. Estimations had close results to tests of the yarns produced, and this helped us to state that these estimation equations can be usable for spinners.

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