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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

Characteristic Properties and Behavior under Flexural and Impact Loadings of Packaging-Waste/E-Glass/Epoxy Sandwich Composites for Construction Applications

Yapı Uygulamaları için Atık-Ambalaj/E-Cam/Epoksi Sandviç Kompozitlerin Eğilme ve Darbe Yükleri altındaki Karakteristik Özellikleri ve Davranışları

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Abstract

Using textile structural composites improves day by day in various industries due to their high specific strength and modulus, good fatigue and corrosion resistance. The most important reasons for the increased use of textile structural composites are the increased expectations for product performance and demand for lightweight materials in global markets. Rapid economic growth, urbanization, rising in population and welfare level lead to an increased amount of waste production. In Turkey, the annual average of solid waste is 25 million tons and 20% of this waste consists of packaging wastes. In this study, a new sandwich material was developed for construction applications with low cost and high performance by using box wastes as core and E-Glass woven fabric as sheet materials. The mechanical properties of sandwich composites were investigated by 3-point flexural test and their impact behavior was compared after low-velocity impact tests in different energy levels. The core material had a negligible flexural strength. The flexural load of sandwich composites was dramatically higher than those of face material. Low-velocity impact results showed that absorbed energy and damaged area increased with increasing impact energy. It could be concluded that the developed sandwich composites absorb more energy with small damaged areas and therefore can be considered as damage resistant materials for constructional applications such as door and siding.

Keywords: Sandwich composites, Packing-waste, E-Glass/epoxy composites, Flexural strength, Impact resistance

Öz

Tekstil yapısal kompozitlerin kullanımı, yüksek özgül dayanım ve modülü, iyi yorulma ve korozyon direncinden dolayı, çeşitli endüstrilerde günden güne artmaktadır. Tekstil yapısal kompozitlerinin kullanımının artmasının en önemli nedenleri, küresel pazarda ürün performansı ve hafif malzemelere olan talebin artmasıdır. Hızlı ekonomik büyüme, kentleşme, nüfus ve refah düzeyindeki artış, atık miktarının da artmasına yol açmaktadır. Türkiye'de yıllık ortalama katı atık miktarı 25 milyon tondur ve bu atığın %20'sini ambalaj atıkları oluşturmaktadır. Bu çalışmada inşaat uygulamaları için, çekirdek olarak atık karton kutu ve dış yüzey malzeme olarak E-Cam dokuma kumaşın kullanıldığı düşük maliyetli ve yüksek performanslı yeni bir sandviç malzeme geliştirilmiştir. Sandviç kompozitlerin mekanik özellikleri 3-nokta eğilme testi ile incelenmiş ve farklı enerji seviyelerinde düşük hızlı darbe testlerinden sonra darbe davranışları karşılaştırılmıştır. Çekirdek malzeme ihmal edilebilir bir eğilme dayanımı göstermiştir. Sandviç kompozitlerin eğilme yükü, dış yüzey malzemelerden çok daha yüksektir. Düşük-hız darbe sonuçları, absorbe edilen enerjinin ve hasarlı alanın artan darbe enerjisiyle birlikte arttığını göstermiştir. Geliştirilen sandviç kompozitlerin daha az hasarlı bölge ile daha fazla enerji absorpladığı belirlenmiş ve bu nedenle kapı ve dış cephe kaplama gibi inşaat uygulamaları için hasara dayanıklı malzemeler olarak kabul edilebileceği sonucuna varılmıştır.

Anahtar Kelimeler: Sandviç kompozitler, Atık-ambalaj/E-Cam/epoksi sandviç kompozitler, Eğilme dayanımı, Darbe direnci.

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I. INTRODUCTION

Textile structural composites have been used in many industrial areas since 1940s, mainly due to developments in thermoset-based polymers, from aerospace to automotive. It is due to the lightness, high strength and modulus, high fatigue resistance and corrosion resistance of composite materials, as well as their production in complex geometric shape parts. [1]. Composites are used extensively in construction industry, from prefabricated houses to bridges [2]. The most used areas of composite materials in construction industry are facade cladding panels and siding materials. Facade claddings can be produced as single-walled or double-walled panels and polyurethane foam can be used for insulating purposes in core part of sandwich composites [3]. Sandwich composites are designed by placing a different material between two layers in a different form. These structures consist of a thick and low density core material between thin and stiff faces. Core materials include foams and aluminum, Nomex and polypropylene (PP) structures in honeycomb or corrugated forms [4]. Honeycomb sandwich constructions are used in where high mechanical strength is required [5, 6]. Some detailed studies were performed by researchers about the mechanical and impact properties of sandwich composites. Cabrea et al. produced sandwich composite by combining PP cores with PP faces in the tubular unit cell structure. It was stated that PP/PP sandwich composites exhibit a more ductile behavior and flexural strengths of these structures are lower compared to E-glass/PP constructions [7]. Xu et al. produced sandwich composites by comprising cores in a wavy beam structure. Sandwich composites showed a face wrinkling-based failure under flexural load which caused by insufficient core resistance [8]. Lascoup et al. developed a sandwich composite that consists of the PU foam core structure with E-glass faces in which the structure is reinforced by an angular stitching through-the – thickness. Stitching density and stitching width are stated as important process parameters [9]. Brandt et al. reported that 3D reinforced sandwich composites exhibit a high potential for damage tolerance and energy absorption capability. The mechanical performance of these materials are influenced by the design parameters. However, it was stated that the crimp caused by weaving process is also an important factor on the mechanical properties [10]. Guan et al. studied both the experimental and numerical effects of stitching on ballistic performance of sandwich composites. The results showed that the stitching had no significant effect on the ballistic strength of the sandwich composites [11]. Meo et al. produced sandwich composites by using Nomex honeycomb core and carbon/epoxy faces. The impact strength of the sandwich composites measured under different energy levels. The energy absorption of Nomex honeycomb was eight-times higher than that of the carbon/epoxy faces [12].

The common feature of commercially produced core materials is their high cost. Nowadays, there is a growing interest in waste-based composites because of the strong need for new low-cost materials which also contribute to recycling. Rapid economic growth, urbanization, rising in population and welfare level lead to an increased amount of wastes. In Turkey, the annual average of solid waste is 25 million tons and 20% of this waste consists of packaging wastes. The amount of packaging placed on the market is 2.5 million tons per year in Turkey. The recycled amount of packaging is about 2 million tons [13]. It could be stated that there is a successful recycling of packaging waste. However, there is not any effort on manufacturing high value added products using these wastes. The novelty of this study is using box wastes as core materials of sandwich structures with E-glass/epoxy faces to obtain low-cost, high-performance and recycled sandwich composites for construction applications.

II. MATERIALS AND METHODS

2.1 Manufacturing of Sandwich Composites

E-glass twill (2/2) woven fabric made from 300 tex fibers was used as face materials of sandwich composites. The areal weight of fabric was 288 g/m^2 and the thickness of fabric was 0.34 mm. The warp/weft density of fabric was 6 ends/10 cm. A corrugated cell type box waste was used as core material. The areal density of box was 0.59 kg/m^2 and the thickness of box was 6.20 mm. Sandwich composites (SC) are produced according to the hand lay-up method using epoxy resin (Hexion, MGS LR160) and hardener (Hexion MGS LH160) in the ratio of 100/25. Curing occurred at 20°C for 24 hours. Figure 1 shows the manufacturing stages of the sandwich composite. For sandwich composite production, the waste box (C) was firstly cut to dimensions of 30x30 cm. E-glass woven fabric are cut in the same dimensions as 4 layers for each faces (F). Teflon films were used on both surfaces for easy releasing of composite. Figure 2 shows the view of produced box waste/E-glass/epoxy sandwich composite. The thickness values of face, core and sandwich composite materials were 1.88 mm, 6.20 mm and 9.73 mm, respectively.

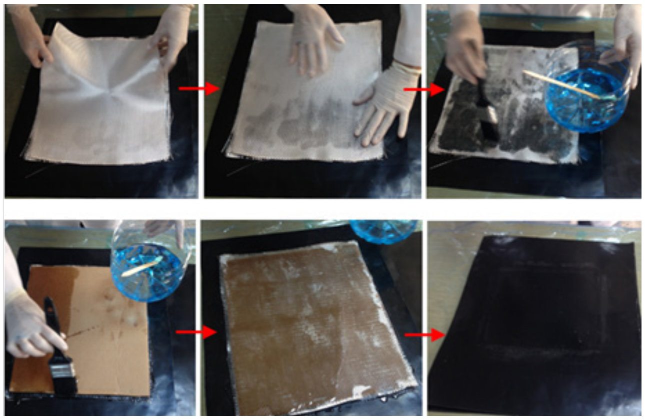

Figure 1. Production stages of the sandwich composite.

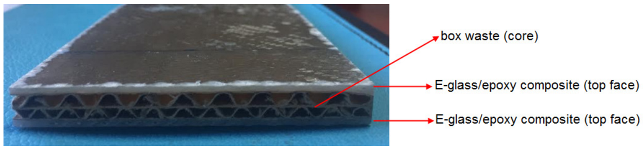

Figure 2. The view of produced box waste/E-glass/epoxy sandwich composite.

2.2 Performed Tests

Density measurements of sandwich composites were performed according to ASTM D792-13 [14] by using a density meter (Precisa®, XP205). The density of the specimens was calculated by measuring the weight of the specimens in the air and in the distilled water at 23±2°C. The composite fiber fraction was measured by burn-off test of ASTM D3171-15 [15] at 650°C for 2 hours. Fiber volume fraction of face composites was calculated by using Equation (1).

$$V_f = [(W/F)/(w/c)] \times 100 \qquad (1)$$

where; V_f is volume based fiber fraction (%), W is fiber weight (g), w is composite weight (g), F is fiber density g/cm^3), c is composite density (g/cm^3).

Flexural properties of sandwich composites were measured according to 3-point flexural test method of ASTM C393-16 [16]. The flexural tests of the sandwich composites were performed on a Zwick-Roell tester. The dimension of the test specimen was 75 mm x 250 mm. The support span length was 200 mm and test speed was 6 mm/min. Figure 3 shows the views of core, face and sandwich composite during flexural test.

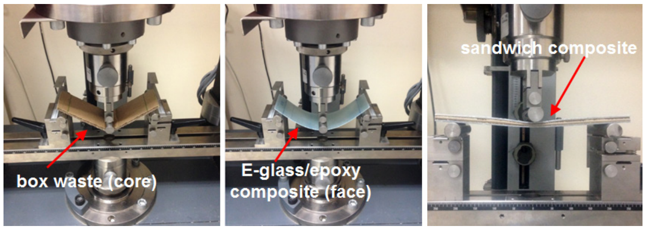

Figure 3. The views of core, face and sandwich composite during flexural test.

Drop-weight impact tests were performed in four different energy levels as 5J, 10J, 20J, 30J and 50J according to ASTM D7136-15 [17] using a CEAST 9350 impact tester. The specimen size was 100 mm x 150 mm. The mass of hemispherical impactor was 5.05 kg and the diameter of impactor was 12 mm. The illustration of impact test machine was given in Figure 4. Peak force and peak deformation values were obtained from impact test. Absorbed energy was calculated by software automatically. After impact test, the front and back face damaged area measurements were conducted on image processing software (BAB Bs200Doc, Turkey). The dent depths of sandwich composites were also measured after impact tests as shown in Figure 4(b).

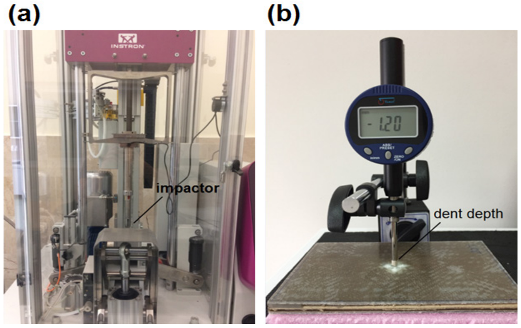

Figure 4. The illustration of impact test machine (a) and dent depth measurement (b).

The flexural strength (2), modulus (3) and strain (4) of sandwich composites were calculated according to the equations of ASTM D790-90 [18] which are given below:

$$S = 3PL/2bd^2 \qquad (2)$$

$$E = L^3 m/4bd^3 \qquad (3)$$

$$e = (l_1 - l_0)/l_0 = \Delta l/l_0 \qquad (4)$$

where; S is the stress in the outer fibers at mid-span (N/m^2), P is the load at a given point on the load-deflection curve (N), L is the support span (m), b is the width of beam tested (m), d is the depth of beam tested (m), E is the modulus of elasticity in flexural (N/m^2), m is the slope of the tangent to the initial straight-line portion of the load-deflection curve (N/m) of deflection. ε is the flexural deflection (%), Δl is the elongation (m) and l0 is the initial length (m).

The core shear strength (5) and facing stress (6) of sandwich composites were calculated according to the equations of ASTM C393-16 [16] which are given below:

$$F_s^{ult} = \frac{P_{max}}{(d+c)b} \tag{5}$$

$$\sigma = \frac{P_{max}\, S}{2t\,(d+c)\,b} \tag{6}$$

where; F_s^{ult} is the core ultimate strength (MPa), P_{max} is the maximum force prior to failure (N), t is the nominal facing thickness (mm), d is the sandwich thickness (mm), c is the core thickness, b is the sandwich width (mm), σ is the facing stress (MPa) and S is the span length (mm).

The absorbed-energy was calculated by software of impact tester according to equation (7):

$$E_A = \frac{1}{2} m \left(v_i^2 - v_r^2\right) \tag{7}$$

where; E_A, is absorbed-energy (J), m is the impactor mass (kg), v_i is the velocity of impact (m/s) and v_r, rebounding velocity (m/s).

III. RESULTS AND DISCUSSIONS

The density, thickness and fiber fraction values of core, face and sandwich composites are presented in Table 1. The thickness of sandwich composite was higher than those of the core and face materials as excepted. The fiber volume fraction of E-glass/epoxy face composite was not high because of using hand lay-up method. And also, the void content of E-glass/epoxy face composite was quite high due to the hand lay-up method. Both fiber volume fraction and void content had obvious effects on the mechanical properties of composites. Density of core material was lower than those of E-glass/epoxy face composite and sandwich composite, as expected. Sandwich composite showed quite low density and areal weight which provide high specific strength.

Table 1. The physical properties of the sandwich composites.

Material type	Thickness (mm)	Density (g/cm^3)	Areal weight (kg/m^2)	Fiber volume fraction (%)	Void content (%)
C	6.20 ± 0.08	0.15 ± 0.03	0.59 ± 0.04	-	-
F	1.88 ± 0.06	1.49 ± 0.09	2.68 ± 0.11	41.40 ± 0.78	5.00 ± 0.492
SC	9.73 ± 0.07	0.62 ± 0.02	6.59 ± 0.16	-	-

The results of flexural and core shear test of sandwich composites are presented in Table 2. Figure 5 shows the load-extension behavior of sandwich composites. As seen in Figure 5, the maximum flexural load of sandwich composite was about 19 times higher than that of core material and about 6.5 times higher than that of E-glass/epoxy face composite. The packaging-waste/E-glass/epoxy sandwich composite was failed by buckling of the panel after flexural load which was generally caused by insufficient panel thickness and core stiffness. E-glass/epoxy face composite showed a more ductile flexural behavior compared to the sandwich composite which confirmed by high deflection. This was probably due to the high support span length of flexural test. However, E-glass/epoxy face composite showed a considerably higher flexural strength compare to sandwich composite. The flexural strength of core material was negligible.

Table 2. Flexural test results and core shear test results of sandwich composites.

Material type	Load (N)	Deflection (%)	Flexural strength (MPa)	Core shear strength (MPa)	Facing stress (MPa)
C	31.20 ± 0.29	180.79 ± 35.45	3.19 ± 0.08	-	-
F	91.88 ± 6.28	2495.96 ± 119.54	103.27 ± 6.51	-	-
SC	587.75 ± 18.32	55.31 ± 3.24	24.70 ± 0.78	0.49 ± 0.01	26.06 ± 1.45

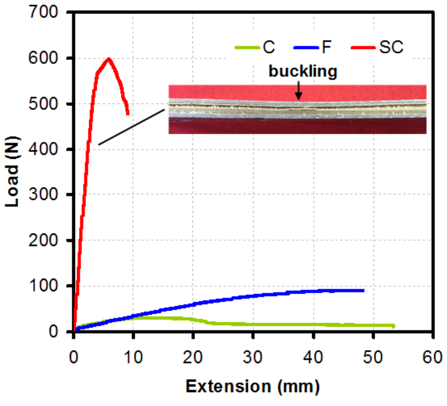

Figure 5. The load-extension behaviour of sandwich composites.

The flexural test results of developed sandwich composite was compared with a produced sandwich composite in literature by using woven glass/PP fabric as face and a 80 kg/m^3 PP honeycomb as core material [7]. The core shear strength of packaging-waste/E-glass/epoxy sandwich composite was 0.49 MPa while the core shear strength of woven glass/PP-honeycomb sandwich composite was 0.14 MPa. These results indicated that the core shear strength of packaging-waste/E-glass/epoxy sandwich composite was about 3.5 times higher than that of woven glass/PP-honeycomb sandwich composite. The facing stresses of packaging-waste/E-glass/epoxy sandwich composite and woven glass/PP-honeycomb sandwich composite were 26.06 MPa and 30 MPa, respectively. Considering the price/performance properties of both sandwich composites, it is possible to achieve the similar performance properties with 30% low-cost by using waste box.

Table 2 presents the impact test results of sandwich composites for 5J, 10J, 20J, 30J and 50J. Figure 6 shows the force-time, force-deformation, energy-time histories of the sandwich composites. Peak force values of sandwich composites were varied from 1783.0 N to 4889.75 N. The peak forces of sandwich composites increased by increasing impact energy levels. At 50J impact energy, the sample was fully perforated. At 30J impact energy, the sandwich composite was partly perforated in which the front face and core were fully perforated while the back face had a severe damage. Since the sandwich composites were perforated at 30J and 50J, their force-time histories were different from those of the 20J, 10J and 5J impacted samples. As seen in Figure 6(a), there were two-peaks were obtained in force-time history of sandwich composites. In peak-1, some matrix-cracks, minor fiber breakages and fiber-matrix delamination were probably occurred. After that the peak force could be reached to its maximum value which defined as peak-2.

Table 5. The impact test results of sandwich composites.

Impact energy	Velocity (m/s)	Peak force (N)	Peak deformation (mm)	Absorbed energy (J)	Damaged area (mm^2)		Dent depth (mm)
					Front face	Back face	
5J	1.43	1783.0	7.53	1.48	90.36	-	0.25
10J	2.02	2466.1	10.22	5.62	189.71	-	0.71
20J	2.85	4507.1	14.78	11.64	288.61	58.29	1.57
30J	3.49	4475.9	16.85	27.79	287.12 (pf*)	214.55 (pf)	5.05
50J	4.51	4889.8	19.29	41.45	267.82 (fp**)	315.24 (fp)	10.73

* pf: partly perforated, ** fp: fully perforated

Peak deformation values of sandwich composites were varied from 7.53 mm to 19.29 mm. Peak deformation values also increased with increasing impact energy levels which indicated that the sandwich composites showed more deflection at higher impact energy levels. The absorbed energy values of sandwich composites were varied from 1.48J to 41.45 J. As shown in Figure 6(c), sandwich composite showed an energy-drop from 5J to 30J energy levels after achieving the peak force because of elastic-recovery. However, there was no an energy-drop at 50J as a consequence of fully perforation that did not give any chance to elastic-recovery of the sandwich composite.

Figure 7(a) shows the damaged areas of sandwich composites on both front and back faces after impact test by considering the peak loads. As seen in Figure 7(a), damaged areas generally increased by the increase in impact energy levels. There were no damaged areas observed on back faces of the sandwich composites at 5J and 10 J impact energy levels while the sandwich composite showed a small back face damaged area at 20 J. The front face damaged areas and peak forces of sandwich composites at 20 J and 30 J impact energy levels were almost same while the sandwich composite showed a 3.7 times higher back face damaged area at 30J. This was due to the partly perforation occurred on front face of sandwich composite at 30J. It can be concluded that the impact damage threshold of sandwich composite was 30J. At this impact energy level, sandwich composite showed a permanent deformation which partly occurred on the front face. Figure 7(b) shows the dent depths of sandwich composites after impact test by considering the absorbed energy. The dent depths of sandwich composites increased with an increase in impact energy levels. The more damage in sandwich composite caused the more energy absorption, as expected.

Figure 8 shows the views of front and back face damaged areas of sandwich composites. Impact load caused multiple-fiber-breakages and severe fiber/matrix delamination on front face. Besides these failures, the fiber pull-out was observed at the back faces of sandwich composites. It can be concluded that the core material restricted the damaged area and increased the energy absorption of the sandwich composites which resulted as relatively damage resistant materials.

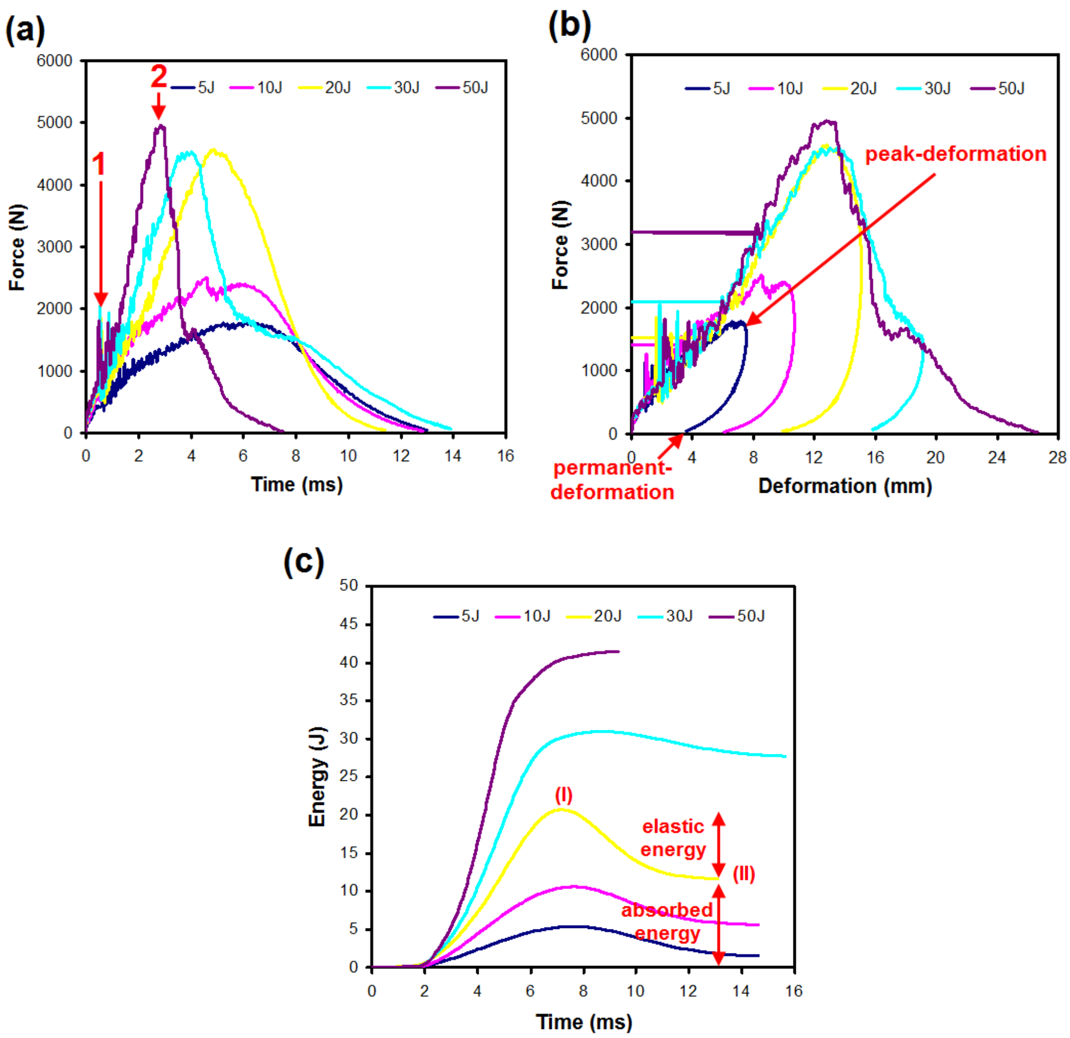

Figure 6. Force-time (a), force-deformation (b), energy-time history (c) of sandwich composites.

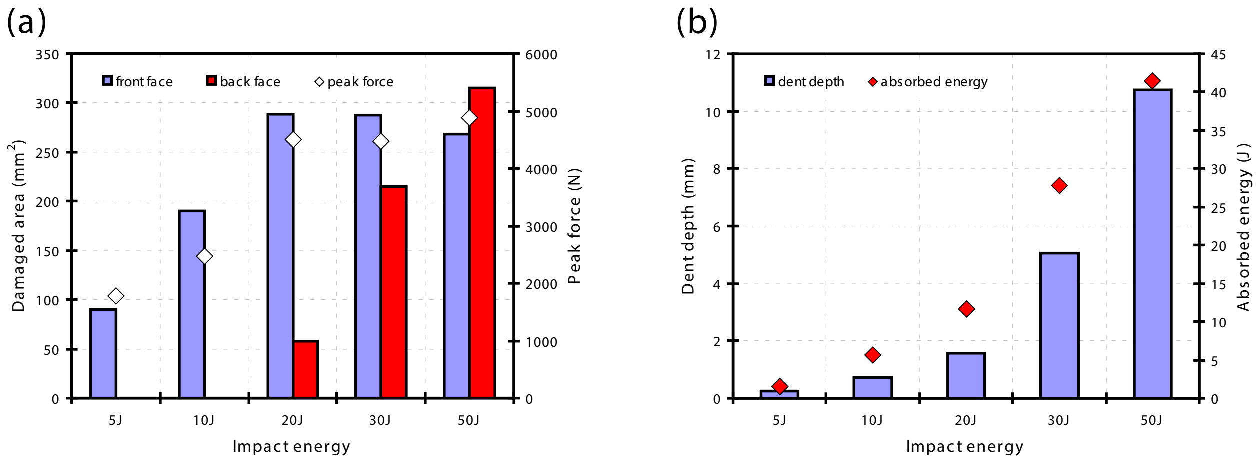

Figure 7. Damaged area-peak force (a), dent depth-absorbed energy (b) of sandwich composites.

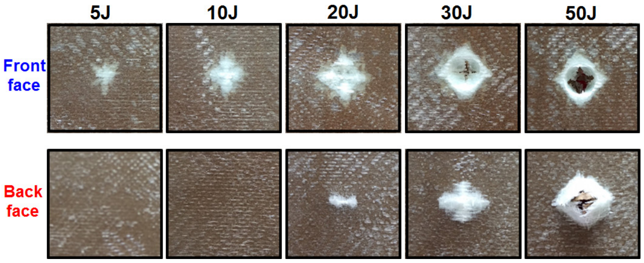

Figure 8. Front and back face damaged area views of sandwich composites.

IV. CONCLUSIONS

A novel packaging-waste/E-glass/Epoxy sandwich composite was developed and its flexural and impact properties were investigated. This sandwich composite had an important contribution to recycling process of packaging wastes in a different perspective. The packaging-waste/E-glass/epoxy sandwich composite showed quite low density and areal weight which provide high specific strength. The compatible flexural test results were obtained compared to literature with quite low-cost. The maximum flexural load of sandwich composite was about 19 times higher than that of the core material and about 6.5 times higher than that of the E-glass/epoxy face composite. The flexural load caused a panel buckling type failure on the sandwich composite which was probably caused by insufficient panel thickness and core stiffness. The peak forces, peak deformations and absorbed energy values of sandwich composites increased with increasing impact energy levels. Because of the fully-perforation at 50J, there was not any elastic-recovery occurred. The damaged areas and dent depths of sandwich composites were generally increased by the increase in impact energy levels, as expected. The more damage in sandwich composite caused the more energy absorption. The impact damage threshold of sandwich composite was determined as 30J in which the sandwich composite showed a partly-perforation on its front face. It can be concluded that the core material restricted the damaged area and increased the energy absorption of the sandwich composites which resulted as relatively damage resistant materials. Using thicker box as core material and filling the cells of box with rigid foams to overcome the buckling type failure and to increase the damage tolerance of sandwich composite could be the subject of future work. By this way, it could be possible to contribute the potential thermal and sound insulation properties of sandwich composites especially using as siding components in constructional applications. The thermal insulation properties of these new sandwich composites will be also investigated in future researches.

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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

Investigation Barrier Properties of Linear Low Density Polyethylene (LLDPE) Spunbond Fabrics

Düşük Lineer Yoğunluklu Polietilen (LLDPE) Spunbond Kumaşların Bariyer Özelliklerinin İncelenmesi

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Abstract

Nonwovens have a growing worldwide market that has a vital proposition in many sectors such as geotextile, medical textile, automotive industry etc. which are supported by technological developments according to the desired characteristics. Among the production methods of the nonwovens, spunbond nonwovens made of polyprophylene (PP) have disposable and durable functional properties and are preferred especially in the field of disposable hygiene textiles. Although the fabric volume market of PP spunbond nonwovens is expected to grow rapidly owing to personal hygiene awareness with an increase in the aging population and regulations for hygiene, polyethylene spunbond nonwovens can probably share the application area due to having a lower melting point and easy processable. In this study, the effect of the weight of linear low density polyethylene (LLDPE) spunbond nonwovens on barrier properties such as resistance to air flow and water were evaluated. Results showed that weight has a statistically significant effect on air permeability and hydrostatic head pressure.

Keywords: Linear low density polyethylene, barrier properties, medical textile, spunbond, nonwoven.

Öz

İstenilen özelliklere gore teknolojik gelişmeler tarafından desteklenen jeotekstil, medikal tekstil, otomotiv endüstrisi gibi birçok sektörde hayati öneme sahip dokusuz kumaşlar dünya çapında büyüyen bir pazara sahiptir. Dokusuz kumaşların üretim metotları arasında polipropilen (PP) spunbond dokusuz kumaşlar tek kullanımlık ve dayanıklı fonksiyonel özelliklere sahiptir ve özellikle tek kullanımlık hijyen tekstilleri alanında tercih edilmektedir. Her ne kadar PP spunbond kumaş pazar hacminin yaşlanan nüfustaki artışı ve hijyen için gerçekleştirilen düzenlemelerle kişisel hijyen bilincinden dolayı hızlı bir şekilde büyümesi beklenirken, polietilen spunbond dokusuz kumaşlar sahip olduğu düşük erime noktası ve kolay ilenebilir olması nedeniyle uygulama alanını paylaşabilir. Bu çalışmada, düşük lineer yoğunluklu polietilen (LLDPE) spunbond dokusuz kumaşların kumaş ağırlıklarının hava ve su geçişine direnç gibi bariyer özellikleri üzerindeki etkisi değerlendirilmiştir. Sonuçlar, kumaş ağırlığının hava geçirgenliği ve hidrostatik su basıncı üzerinde istatistiksel olarak önemli bir etkisi olduğunu göstermiştir.

Anahtar Kelimeler: Düşük lineer yoğunluklu polietilen, bariyer özellikleri, medical tekstil, spunbond, dokusuz kumaş.

I. INTRODUCTION

Nonwoven fabrics are technical textiles manufactured using sheets or webs bonded via various thermal, chemical or mechanical processes. Such fabrics can be produced or modified to exhibit superior properties in accordance with the requirements of particular applications [1]. Nonwovens play an important role in the application of medical textiles. The use of PP as raw materials has a vital for hygiene and medical sector such as baby and adult diapers, medical products, feminine care etc. Figure 1 illustrates the PP nonwoven fabric market volume share in 2015 according to the product and application area. In this figure, PP spunbonded nonwovens have the highest production rates and hygiene and medical sectors have predominant volume and it is obvious that in these areas there are probably higher potential to investigate.

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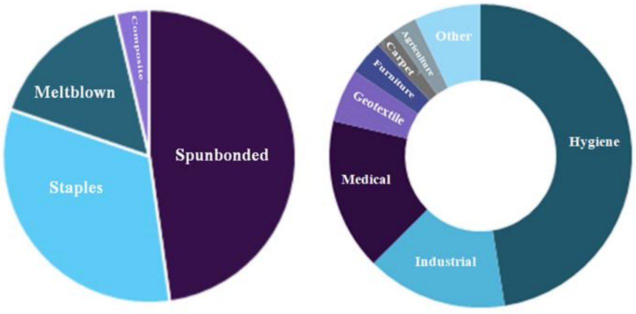

(a) (b)

Figure 1. PP nonwoven fabric market volume share in 2015 (a) by product and (b) by application [2,3].

Polyethylene (PE) comes under the general category of polyolefins as the same as PP fiber and is also used in the nonwoven application. It can be classified as PE fibers into high density polyethylene (HDPE) and linear low density polyethylene (LLDPE). But there has been limited research on PE fibers [4]. Spunbond PE has an advantages that are lightweight, easy processing, good chemical resistance, good impact strength, excellent electrical properties, good barrier properties, low water absorption, toughness, and flexibility even at extremely low temperature, safe for food contact and non-toxic for skin contact, do not require pre-drying and recyclable [5]. Also, lower working temperature is needed than polypropylene polymer and LLDPE polymer has low moisture absorption rates [6] and it gives a softer fabric [7]. This study was focused on the evaluation of barrier properties in a different weight of LLDPE spunbonded nonwovens.

II. MATERIAL AND METHOD

In this study, LLDPE was chosen as the raw material of spunbonded nonwovens production because of the lower temperature necessary to processability than HDPE ones. The melt flow index of raw material was determined at 190°C temperature under 2.16 kg applying load as 11.6 g/10 min value. Nonwovens in different weight were manufactured in spunbond process which the schematic representation is shown in Figure 2.

Before the production, several trials were conducted to adapt the machine parameters for LLDPE spunbonded nonwoven. After these trials, optimum parameters were determined and the machine production settings as a range were given in Table 1.

Table 1. Main production settings.

Production Settings	Value
Extrusion Temperature,°C	210-250
Die Temperature,°C	200-240
Calendar Speed,m/min	50-300
Calendar Bonding Temperature,°C	135
Nip Point Pressure,bar	80-90

Before tests, LLDPE spunbonded nonwovens with 40 gsm, 50 gsm, 60 gsm, 80 gsm, and 100 gsm weights were conditioned at 20 ± 2 °C and 65 ± 4 % for 24 hours. The air permeability of samples was conducted according to the WSP 70.1 (05)-Standard Test Method for Air Permeability of Nonwoven Materials standard at 200 Pa pressure drop by using 20 cm^2 test area by means of Sdl Atlas air permeability tester. Ten tests were performed for each sample and the mean results of air permeability in mm/sec were evaluated. The hydrostatic pressure of samples was determined by means of Sdl Atlas hydrostatic head tester in accordance with WSP 80.6 (05) Standard Test Method for Evaluation of Water Resistance (Hydrostatic Pressure) Test standard. In this test, the rate of increase of water pressure was chosen as 60 cmH_2O/min by using 100 cm^2 test head. Distilled water at 23 ± 2 °C temperature was used and tests were completed when three separate drops are formed on the different surface regions of the specimen. Five tests were performed for each sample. Hydrostatic pressure values were obtained as a cmH_2O unit.

In order to evaluate the effect of weight on air permeability and hydrostatic pressure statistically, variance analysis was performed by means of the SPSS package program at a 95% confidence interval. Furthermore, the significant difference between weights was determined with post-hoc Tukey HSD multiple comparison test analysis.

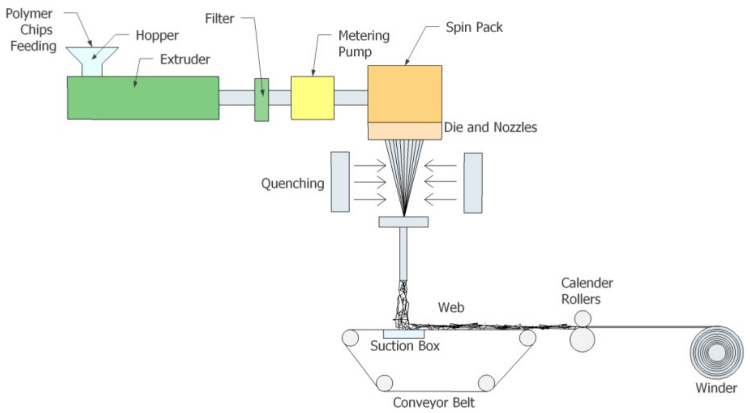

Figure 2. Schematic representation of spunbond nonwoven production.

III. RESULTS AND DISCUSSIONS

The physical properties of LLDPE spunbonded nonwovens are given in Table 2.

Table 2. Physical properties of LLDPE spunbonded nonwovens.

LLDPE Spunbonded Nonwoven Samples	Thickness, mm	Weight, gsm
40	0.30	40.3
50	0.35	50.1
60	0.41	61.3
80	0.56	80.2
100	0.67	101.1

The air permeability of LLDPE spunbonded nonwovens was illustrated in Figure 3. It is obvious that an increase in the weight of the nonwovens affects the resistance to air flow positively by increasing the fiber volume within the nonwoven structure that prevents the air flow from the one side of the nonwoven to another. To produce the lower weight spunbonded nonwovens, changed machine settings affect the fiber thickness increase finally higher pore size arises. The best barrier property to air flow was obtained in 100 gsm LLDPE spunbonded nonwoven. The thickness of nonwovens also affects the air flow that is nonwoven with higher thickness show higher resistance.

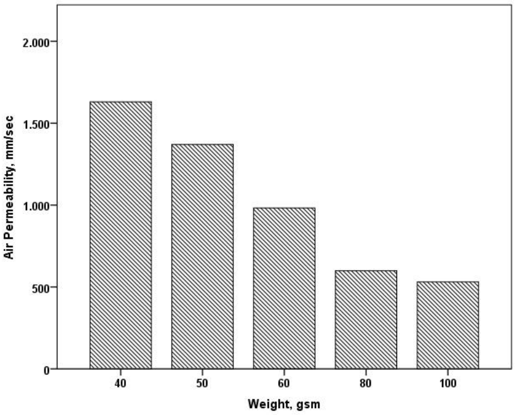

Figure 3. Air permeability of LLDPE spunbonded nonwovens.

The resistance to the water of nonwoven samples is shown in Figure 4. Hydrostatic pressure in cmH_2O of LLDPE spunbonded nonwovens shifts positively from lower weight to higher one that makes the nonwoven more resistance to water pressure. It can be said that 40 gsm, 50 gsm and 60 gsm of LLDPE spunbonded nonwovens show similar resistance to water pressure because of the lower or 10 gsm differences between these nonwovens. On the other hand, resistance to the water pressure of 60 gsm is lower than 50 gsm nonwoven but the difference is low so it can be ignored. When 80 gsm and 100 gsm nonwovens hydrostatic pressure are taken into consideration, these are also similar resistance and higher than that of three ones. Thickness and weight of the nonwovens are related to each other that make the nonwoven more compact and have a lower pore size. Under the pressure, water can easily come out to the surface of the nonwoven with higher pore size.

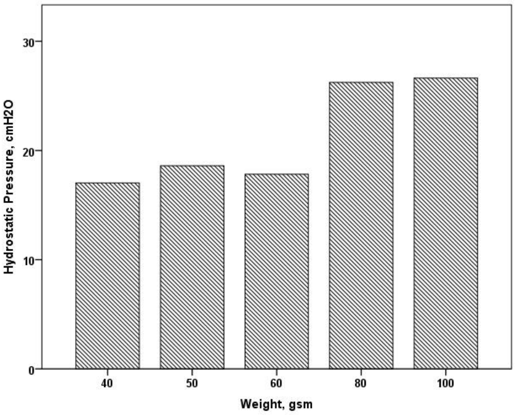

Figure 4. Hydrostatic pressure of LLDPE spunbonded nonwovens.

Statistical analysis results were shown in Table 3. According to the analysis of variance results, the effect of weight on air permeability was found to be statistically significant with $p<0.001$. The analysis explains about 97.8 % of the variability in air permeability for all nonwovens. Besides, weight has also statistically significant ($p<0.001$) effect on hydrostatic pressure and adjusted R^2 was determined as 94% with a very high value.

Table 3. Analysis of variance of LLDPE spunbonded nonwovens with different weight.

Source	Dependent Variable	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	**Air permeability**	2890140.933[a]	4	722535.233	158.402	0.000	0.984
	Hydrostatic pressure	270.867[b]	4	67.717	55.658	0.000	0.957
Intercept	**Air permeability**	16159584.067	1	16159584.067	3542.681	0.000	0.997
	Hydrostatic pressure	6784.067	1	6784.067	5575.945	0.000	0.998
Weight	**Air permeability**	2890140.933	4	722535.233	158.402	0.000	0.984
	Hydrostatic pressure	270.867	4	67.717	55.658	0.000	0.957
Error	**Air permeability**	45614.000	10	4561.400			
	Hydrostatic pressure	12.167	10	1.217			
Total	**Air permeability**	19095339.000	15				
	Hydrostatic pressure	7067.100	15				
Corrected Total	**Air permeability**	2935754.933	14				
	Hydrostatic pressure	283.033	14				
a. R Squared = 0.984 (Adjusted R Squared = 0.978)							
b. R Squared = 0.957 (Adjusted R Squared = 0.940)							

Table 4 displays the Tukey HSD multiple comparison results of LLDPE spunbonded nonwovens. It can be said that there is no difference between of air permeability of 80 gsm and 100 gsm nonwovens. From 100 gsm to 40 gsm nonwovens air permeability increases. The highest air permeability was obtained from 40 gsm nonwoven with high pore size. In Table 5, Tukey HSD multiple comparison results show that there is no such difference between 40 gsm, 50 gsm and 60 gsm nonwovens for hydrostatic pressure. Likewise, 80 gsm and 100 gsm nonwovens were found as the same.

Table 4. Tukey HSD multiple comparison for air permeability.

Weight, gsm	Subset			
	1	2	3	4
100	548.33			
80	587.67			
60		997.00		
50			1390.00	
40				1666.67
Sig.	0.949	1.000	1.000	1.000
The error term is Mean Square(Error) = 4561.400.				
Uses Harmonic Mean Sample Size = 3.000.				
Alpha = 0.05.				

Table 5. Tukey HSD multiple comparison for hydrostatic pressure.

Weight, gsm	Subset	
	1	2
40	17.033	
60	17.833	
50	18.600	
80		26.233
100		26.633
Sig.	0.454	0.991
The error term is Mean Square(Error) = 1.217.		
Uses Harmonic Mean Sample Size = 3.000.		
Alpha = 0.05.		

IV. CONCLUSIONS

As a conclusion, LLDPE spunbonded nonwovens are easily used with an alternative to the PP spunbonded nonwovens when required properties obtained according to the application areas. In the medical sector, when talking about the surgical clothes these nonwovens can be used with softer fabric properties which makes it more comfortable especially than PP spunbonded nonwoven. With optimum machine settings, LLDPE spunbonded nonwovens can be easily produced. This study was evaluated the barrier properties of LLDPE spunbonded nonwovens against air flow and water pressure. Thus, five weight types of spunbonded nonwovens were produced as one layer. Results showed the effect of the weight on air permeability and hydrostatic pressure was found to be statistically significant.

For further study, it is planned to investigate the LLDPE spunbonded nonwovens with LLDPE film coating at different production parameters. Besides, PP spunbonded nonwovens with LLDPE film coating will be also manufactured in order to compare the properties.

Acknowledgements

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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

The Investigation of Antimicrobial Properties of Ag–Alginate Impregnated Polyester/Viscose Nonwoven Fabric

Ag – Aljinat Emdirilmiş Polyester / Viskon Nonwoven Kumaşın Antimikrobiyal Özelliklerinin Araştırılması

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Abstract

Alginate is a biomaterial that is used in food, textile and medical industry. Alginate is used in textile industry as sizing agent, thickener and finishing materials.In addition to this, it is used in medical industry due to its biodegradable and biocompatible properties. The aim of this study is to investigate the antimicrobial properties of Ag–alginate impregnated polyester/viscose nonwoven fabric. In this study, Ag–alginate solutions were prepared. Polyester/viscose nonwoven fabric with different mixing ratio was impregnated with prepared solutions. Finally, The antimicrobial, EDX and SEM analyses were performed.

Keywords: Ag–alginate, Antimicrobial Properties, Polyester/Viscose Nonwoven

Öz

Aljinat, gıda, tekstil ve tıp endüstrisinde kullanılan bir biyomateryaldir. Aljinat tekstil endüstrisinde haşıl maddesi, kıvam arttırıcı ve terbiye maddesi olarak kullanılmaktadır. Buna ek olarak biyobozunur ve biyouyumlu özellikleri nedeniyle medikal endüstride de kullanılmaktadır. Bu çalışmanın amacı, Ag-aljinat emdirilmiş polyester / viskon nonwoven kumaşın antimikrobiyal özelliklerini incelemektir. Bu çalışmada Ag-aljinat çözeltileri hazırlanmıştır. Farklı karışım oranlarına sahip polyester / viskon dokunmamış kumaşlar hazırlanan çözeltilerle emdirilmiştir. Son olarak, antimikrobiyal, EDX ve SEM analizleri yapılmıştır.

Anahtar Kelimeler: Ag-Aljinat, Antimikrobiyal Özellikler, Polyester/Viskon Nonwoven

I. INTRODUCTION

Alginate (alginic acid sodium salt) biopolymer obtained in brown seaweed; a polysaccharide consisting of guluronic and mannuronic acid monomers [1]. Alginate has biocompatible and biodegradable properties [2]. Alginate has been utilized in a variety of applications such as food additives, pharmaceuticals, cosmetics and textile manufacturing. In textile ındustry, alginate is used as a sizing agent, thickener and finishing materials [3]. Besides, İt is used to produce medical textile materials and fibers such as calcium alginate, sodium alginate and silver alginate fibers. Alginate keeps the humidity level of the micro environment constant, reduces the wound infection, and facilitates wound healing [4]. Therefore, it is used as a wound dressing material.

In terms of human health, microbial contamination of textile products used for medical purposes in hospitals is important [4]. For this reason, antimicrobial textile products are preferred in hospitals [5]. Different antibacterial agents such as quaternary ammonium compounds, triclosan, chitosan, natural-based antimicrobial agents, metals and metallic salts, poly

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(hexamethylene biguanide) (phmb), regenerable n□halamine, are used in order to give antibacterial properties to textile products [6]. In particular, studies have predominantly being made to gain antibacterial properties with silver in textile industry. It has been known for 200 years that silver is healing wounds [4]. Controlled release is an important part of work done with silver ions, so, many studies have been conducted in relation to controlled release [7].

The objective of this study is to investigate the antimicrobial properties of Ag–alginate impregnated polyester/viscose nonwoven fabric. In this study, silver nitrate, sodium alginate and calcium chloride solutions were prepared. Secondly, samples were impregnated with the calcium chloride and silver nitrate solution then impregnated with sodium alginate. Finally, the antimicrobial, EDX and SEM analyses were performed.

II. EXPERIMENTAL SET-UP AND PROCEDURE

2.1 Materials

In this study polyester/viscose nonwoven fabric with different mixing ratio were used to be impregnated with prepared solutions. The ratio of polyester/viscose fiber was three different quantities. First of the fiber ratio was 80% viscose to 20% polyester, second of the fiber ratio was 70% viscose to 30% polyester, third of the fiber ratio was 40% viscose to 60% polyester.

Sodium alginate (E401) with 500-600 cps viscosity and 80 mesh high molecular weight and povidone-iodine (PVP-I) was purchased from Kimbiotek (Turkey). Silver nitrate with 4.35 g/cm^3 (20 °C) density, 212 °C melting point and 169.87 g/mol molar mass was purchased from Merck. Calcium chloride with 15 – 30mm approx. size, 110.99 gr molecular weight was purchased from Merck surechem products.

2.2 Method

2.2.1 Preparation of Samples with $AgNO_3$

In the beginning of application solutions used were prepared. The preparation of Na-Alg matrix is quite directly forward.

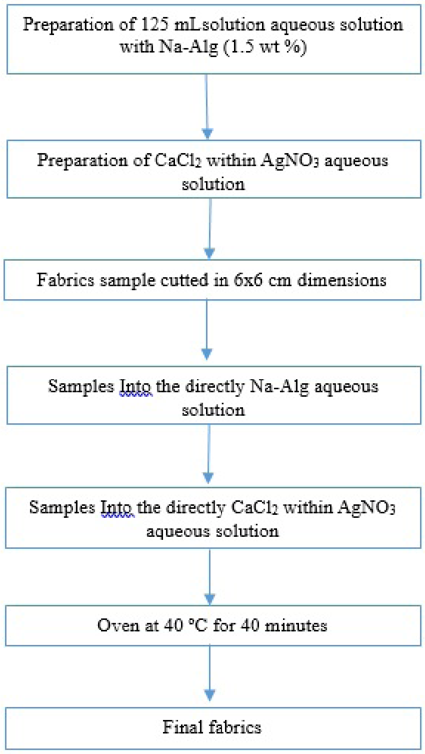
In a typical synthesis 125 mL of Na-Alg (1.5 wt %) was added to a 125 mL aqueous solution. Following that, exactly 1.87 gr. Na-Alg was mixed in the totally 125 ml aqueous solution at 70 °C for 100 minutes in a 1600 rpm. Together with this process, on the other side $AgNO_3$ (0.05 M) solution were prepared. According to the solution preparation process, 1.06 gr $AgNO_3$ (0.05 M) particul was added to 125 ml aqueous solution. Then, it was mixed in the solution until the particles were completely dissolved in solution [8].

Furthermore provided, $CaCl_2$ solutions were prepared to get a stronger applique to nonwoven fabric. Following the prepartions of solutions, $CaCl_2$ solution was added to $AgNO_3$ aqueous solution and both were mixing until getting a homogenean mixture. It provided improvement of adhesion between solutions on the fabrics. In this manner, an effective finishing process was performed.

After solutions were obtained ready to apply to sample, fabrics were cutted in 6x6 cm dimensions. In this way, totally 9 sample were prepared for application. Three of them were 80% viscose to 20% polyester fabric, three of which were for SEM analyises, three of which were for EDX analyses and the last three of which were for the antimicrobial analyses. Nine of them were 70% viscose to 30% polyester fabric and the last nine of them were 40% viskon to 60% polyester for the same analyses. At the end of this process there were 9 samples which were impregnated solutions to perform SEM, EDX antimicrobial analyses.

Samples were firstly plunged into the Na – Alg solution, after this process they were immediately plunged into the $AgNO_3$ solution with $CaCl_2$ inside it sequentially. After impregnating process, they were put in to the oven at 40 ºC and for 40 minutes to get them dry and finished for analyses performing.

Table 1. Work-flow of preparation of samples with $AgNO_3$



2.2.2 Preparation of Samples without $AgNO_3$

As in the beginning of application solutions used were prepared. First, 125 mL of Na-Alg (1.5 wt %) was added to a 125 mL aqueous solution as it had been applied at first process. Following that, exactly 1.87 gr. Na-Alg was mixed in the totally 125 ml aqueous solution at 70 °C for 100 minutes in a 1600 rpm. Afterward, three pieces of each type fabric for all analyses were prepared with only Na-Alg subsequently $CaCl_2$ applicated without any $AgNO_3$ to obtain the antimicrobial effect of $AgNO_3$ by comparison between these two type of applied samples. After this application process, there are 9 samples without $AgNO_3$ and totally 18 samples. Also, there were getting 9 untreated raw fabrics for using them as reference sample to make comparision between them and applique fabrics.

Finally, it has been totally 27 samples to perform the antimicrobial, EDX, SEM analyses of the each other. The list of the samples number and properties is given below.

Table 2. The number of fabric samples to use in analyses

Fabric (x3)	Fabric Content	Applied Chemical
1	80% viscose to 20% polyester	None
2	70% viscose to 30% polyester	None
3	40% viscose to 60%polyester	None
4	80% viscose to 20% polyester	Na-Alg + $CaCl_2$
5	70% viscose to 30% polyester	Na-Alg + $CaCl_2$
6	40% viscose to 60% polyester	Na-Alg + $CaCl_2$
7	80% viscose to 20% polyester	Na-Alg + $CaCl_2$ with $AgNO_3$
8	70% viscose to 30% polyester	Na-Alg + $CaCl_2$ with $AgNO_3$
9	40% viscose to 60% polyester	Na-Alg + $CaCl_2$ with $AgNO_3$

III. ANALYSIS

3.1 Physical and chemical properties of samples

Physical and chemical structures of samples were analyzed with SEM and EDX by using ZEISS/EVO 40 Electron Microscope.

Electron Microscopy instruments (Scanning Electron Microscopy (SEM) where the imaging capability of the microscope identifies the specimen of interest was analyzed. The data generated by EDX analysis consist of spectra showing peaks corresponding to the elements making up the true composition of the sample being analysed. Elemental mapping of a sample analysis were also presented.

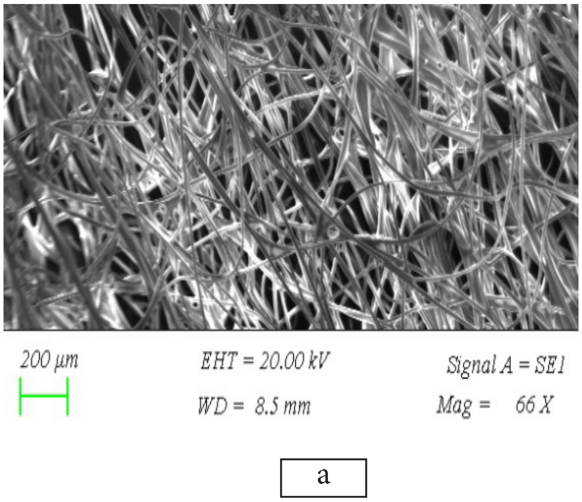
3.2 Antibacterial Analysis

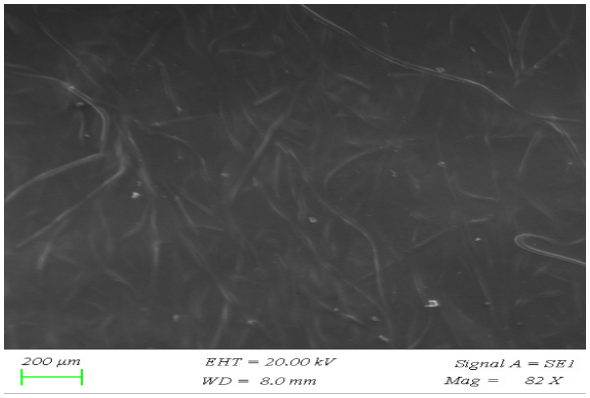
For the AATCC 100 method, a square sample of 4.8×4.8 cm was used. In the AATCC 100 method, an inoculum with 100 ml of LB and incubated for 24 h at 37±1°C was used. Its bacteria concentration was adjusted with LB 10^5 cells ml^{-1}. Then, 1±0.1 ml of the diluted inoculum was placed in each sample. 50 ml sterile distilled water was added and the sample 1.This was vigorously shaken for 1 minute. 100 µL of the liquid sample was placed on LB agar plate. This represents “zero contact time”. The other samples were incubated for 24 h at 37±1°C. After the incubation period, 50 ml sterile distilled water was added and was vigorously shaken for 1 minute. 100 µL of the liquid sample was placed on LB agar plate. This represents “24 contact time”. All plates were incubated 37±1°C for 24 hours. After incubation, colonies of recovered were counted used to determine percent reductions. In this study, *Staphylococcus aureus* (Gram positive) and *Klebsiella pneumoniae* (Gram negative) were used as test organisms.

IV. RESULTS AND DISCUSSIONS

4.1 Results of SEM

SEM of sample 7, sample 4 and sample 1 are given in figure 1.





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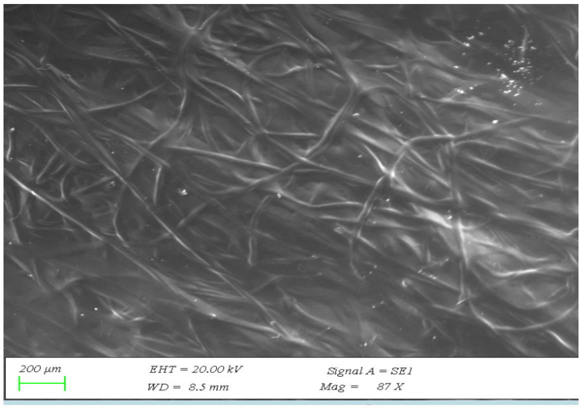

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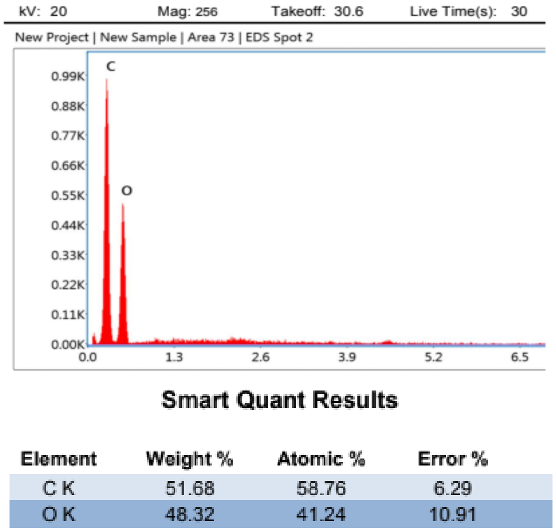
Figure 1. SEM results of (a) sample 1, (b) sample 4 and (c) sample 7

SEM images show images of the untreated sample and the alginate $CaCl_2$ sample and the silver sample with 80/20 ratio of viscose/polyester. The reason why only 80-20% of fabrics treated with $AgNO_3$ in SEM images were given is that, it is due to the excess of the silver element in the fabric.

According to the normal sample, sample with $CaCl_2$-alginate appears to be cross-linked on the surface due to calcium. Silver particles in the sample with Silver alginate $CaCl_2$ is visible as small microparticles on the surface, as it can be seen from the SEM images.

4.2 Results of EDX analysis

Edx analysis gave the content of elementes in the chemical structure of materials. Totally, nine different fabric samples were analyzed in EDX instrument (ZEISS/EVO). When references samples were investigated, it was seen there were C and O elements were presented and while ratio of viscose in the fabric was increasing, quantity of O element was rising. Afterwards, samples with only Na-Alg and $CaCl_2$ applied were investigated. Unlike the first samples group, extra diffrent elements were appeared as Cl, Ca, Na, and K elements. Relying on this result, it can be certainly said that Na-Alg and $CaCl_2$ solutions successfully appliquéd onto the samples. EDX graphs and table were given below to compare the EDX results of samples belonged to two diffrent groups.



Smart Quant Results

Element	Weight %	Atomic %	Error %
C K	51.68	58.76	6.29
O K	48.32	41.24	10.91

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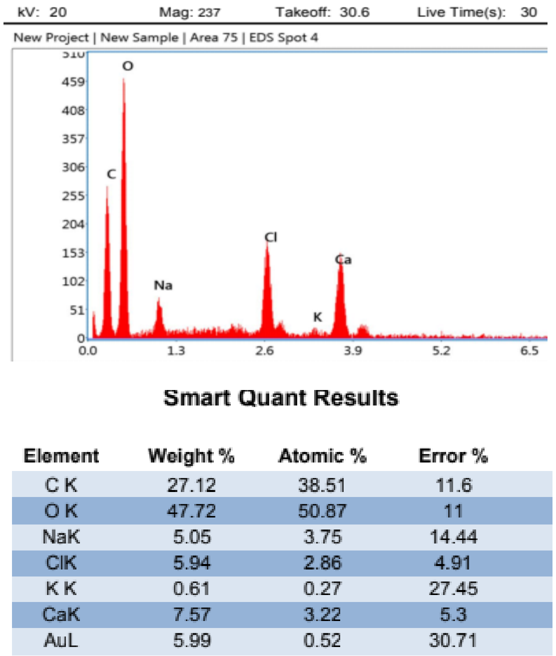


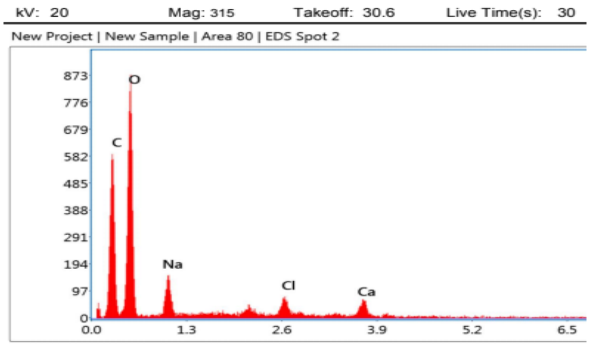
Smart Quant Results

Element	Weight %	Atomic %	Error %
C K	27.12	38.51	11.6
O K	47.72	50.87	11
NaK	5.05	3.75	14.44
ClK	5.94	2.86	4.91
K K	0.61	0.27	27.45
CaK	7.57	3.22	5.3
AuL	5.99	0.52	30.71

b

Figure 2. EDX results of (a) reference and (b) NaAlg-$CaCl_2$ applied samples

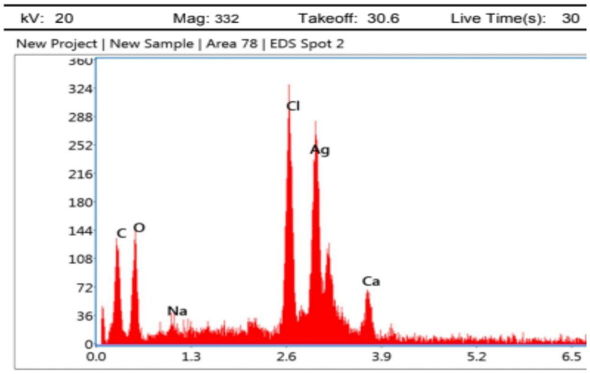
Finally, EDX results of samples appliquéd with $AgNO_3$ were investigated. According to edx results, there were not seen any Ag ions onto the samples with 40% viscose to 60% polyester content. However, as opposed to this result, onto the samples with 70% viscose to 30% polyester content and samples with 80% viscose to 20% polyester content there were seen Ag element ions in different levels. The high level of Ag ions was presented onto the sample with 70% viscose to 30% polyester content. Approximately to that, sample with 80% viscose to 20% polyester content contains high level of Ag ions in its chemical structıre. It can be said from these results, the Ag ion is more easily trapped in the fabric, if viscose ratio is more than polyester ration in the fabric content. So that, it was decided that samples with 80% viscose to 20% polyester content must be analysed for the anti-microbial characteristic as they have Ag ions in their structure and they have antimicrobial protective properties. Edx graphs and element lists of three types of samples were given below. As it is shown from the graphs and lists of elements, only samples which have more viscose contents than polyester contens have Ag ions on them.



Smart Quant Results

Element	Weight %	Atomic %	Error %
C K	34.11	44.41	9.1
O K	50.21	49.08	10.18
NaK	6.67	4.54	11.59
ClK	1.59	0.7	10.82
CaK	2.19	0.85	9.4
AuL	5.23	0.42	34.49

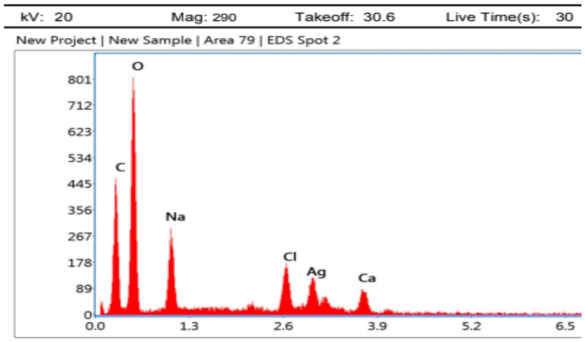
a



Smart Quant Results

Element	Weight %	Atomic %	Error %
C K	20.87	39.1	14.56
O K	29.04	40.85	13.7
NaK	3.04	2.98	25.35
ClK	13.34	8.47	5.04
AgL	29.25	6.1	6.5
CaK	4.46	2.5	16.46

b



Smart Quant Results

Element	Weight %	Atomic %	Error %
C K	25.9	37.57	10.27
O K	45.41	49.45	10.33
NaK	11.61	8.8	10.56
ClK	3.35	1.64	7.92
AgL	5.52	0.89	8.97
CaK	2.66	1.16	9.97
AuL	5.54	0.49	31.12

c

Figure 3. EDX results of (a) 40% viscose to 60% polyester, (b) 70% viscose to 30% polyester, (c) 80% viscose to 20% polyester

4.3 Results of Antibacterial analysis

Antibacterial activity of sample 7, sample 4 and sample 1 are given in table 3.

Table 3. Antibacterial activity of sample 7, sample 4, sample 1.

Sample	Bacteria	Antibacterial Activity (%)
7	*S. aureus*	100
	K. pneumoniae	100
4	*S. aureus*	Increase in the number of bacteria
	K. pneumoniae	Increase in the number of bacteria
1	*S. aureus*	No bacterial growth
	K. pneumoniae	No bacterial growth

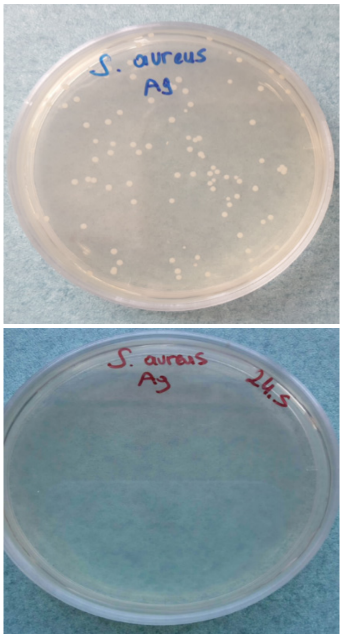


Figure 4. Bacterial growth in a (zero contact time) and b (24 contact time) for Sample 7 on *S. aureus*

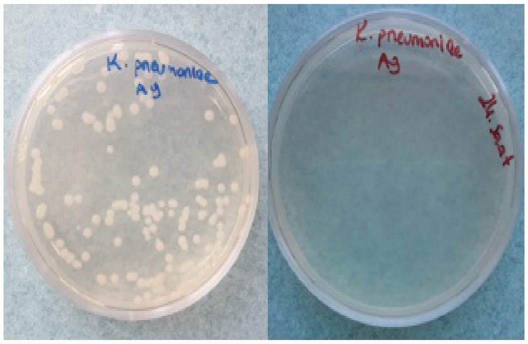


Figure 5. Bacterial growth in a (zero contact time) and b (24 hours contact time) for Sample 7 on *K. pneumoniae*

Regarding the results obtained with the AATCC 100 method, it is possible to notice that samples 7 presented 100% of growth inhibition for both bacteria, while sample 4 presented increasing in the number of bacteria for *S. aureus* and *K. pneumoniae*. Otherwise bacterial growth was not observed in sample 1. In sample 4, bacteria consumed the alginate as food source, in sample 1 conditions was not suitable for bacteria growth.

V. CONCLUSIONS

As a result of the SEM and EDX analyses, calcium alginate formation was observed on nonwoven surfaces containing viscose and polyester at different ratios where alginate and calcium chloride were applied. In addition, antibacterial activity was not observed in samples containing calcium alginate. The absence of antibacterial activity is thought to be due to the absence of structure that would prevent antibacterial activity in the structure and alginate to be formed by nutrients for bacteria.

In the samples treated with $AgNO_3$-$CaCl_2$-Alginate, it is difficult to substitute the sodium element in the sodium alginate structure of silver, making the formation of silver alginate difficult [3]. For this reason, in order to achieve formation of silver surface in the study, $AgNO_3$ and $CaCl_2$ were processed together. As a result of the analysis, the increase of the viscose ratio and the increase of the silver ratio on the surface after the application were thought to be caused by the functional groups in the viscose structure. Antibacterial activity was observed on the surfaces due to silver formation.

Finally, in the first antibacterial test made on the untreated reference fabric, no bacteria were found in the test made after 24 hours when the bacteria were growing. This is thought to be due to the fact that they can not sustain their nutrient-free life for 24 hours at 37 ° C.

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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

Printability of Bamboo Jersey Fabrics in Thermal Transfer Printing System

Bambu Süprem Örme Kumaşların Termal Transfer Baskı Sistemindeki Basılabilirliği

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Abstract

Thermal transfer printing system, which works transferred onto textile fabric by heat of the image formed in the film material surface, is a printing system used in textile industry. The exactly transferring onto the textile fabric of the image on the film material and the remain stable of the transferred image after the use and washing conditions are an important indication for a quality print. In this study, firstly it was determined the physical properties of the bamboo jersey fabrics. Then, it was transferred of the image in the thermal printing system on the fabrics at 3 different temperatures. And then it was performed the washing process of fabrics before and after printing and measured the values of L*a*b*, the gloss and the light resistance of the printed fabrics. From the obtained data, the printability of the bamboo jersey fabrics in the thermal printing system and the resistance of the print after the standard washing were determined.

Keywords: Bamboo, Jersey, Thermal print, Printability

Öz

Film malzeme yüzeyinde oluşturulan görüntünün, ısı yoluyla tekstil kumaşları üzerine aktarılması yöntemiyle çalışan Termal Transfer Baskı Sistemi Tekstil sektöründe de kullanılan bir baskı sistemidir. Film malzeme üzerindeki görüntünün kumaş üzerine tam olarak aktarılması ve aktarılan bu görüntünün kullanım şartlarına ve yıkama süreçleri sonrasında stabil kalması kaliteli bir baskının önemli göstergeleridir. Çalışmada ilk olarak bambu süprem örme kumaşların fiziksel özelliklerinin belirlenmiş ve sonra kumaşlar üzerine 3 farklı sıcaklıkta termal baskı sisteminde görüntünün aktarılmıştır. Baskıdan önce ve sonra kumaşların yıkama işleminin gerçekleştirilmesi ve kumaşlara basılmış renklerinin L*a*b*, gloss ve ışığa karşı direnç değerlerinin belirlenmesi olmak üzere dört aşamalı olarak gerçekleştirilmiştir. Elde edilen verilerden, bambu süprem örme kumaşların termal baskı sistemindeki basılabilirlikleri ve baskının standart yıkanma sonrasındaki dayanımları belirlenmiştir.

Anahtar Kelimler: Bambu, Süprem örme, Termal baskı, Basılabilirlik

I. INTRODUCTION

The thermal transfer printing system is an environmentally friendly printing system that will not leave the solvent vapor harmful to the environment and the human body [1]. It is a printing system in which image transfer is carried out by using the method of adhering the pigments on the film material to the substrate under pressure with the use of temperature and pressure [2].

Heat, pressure and applied pressure duration are important pressure parameters that determine print quality in this printing system. Also, these printing parameters, which are determined according to the surface properties of the substrate, are important for high print quality [3-6].

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In thermal transfer printing systems, image on transfer papers are transferred by the screen printing or digital technology methods [7]. The solid paint particles on the transfer paper are transferred to the substrate after the heat and pressure formed during printing [8].

The temperature and the applied time period in thermal transfer printing is vary according to the substrate. In this study, it was made for determining the effect on the print quality and durability of the image transferring onto bamboo jersey fabrics at different temperatures in the thermal transfer printing.

II. MATERIALS AND METHODS

2.1. Textile Fabric Properties

In this study, bamboo jersey fabrics were used. The fabric structure is with yarn No Nm30, Twist z 402x2, 490x2, 407x2, 419x2, 401x2, grammage 1.305 g/m^2.

2.2. Creating Transfer Printing Film

Printing master films were prepared and then they were printed on polyester film by HP Indigo 5500 digital printing machine. In order to prevent spreading of the image, the surfaces of printed polyester films were coated with white dye using a semi-automatic screen-printing machine. The mesh number was 140 threads/cm (tpc). After print, they were dried at 60°C. And then, it was coated with water-based adhesive for giving binder feature using screen-printing system. The mesh number was 68 threads/cm (tpc).

2.3. Carrying out Thermal Transfer Printing

The obtained printing master films were transferred on cotton, polyamide and polyester textile fabrics using Sahok Sh 49BD thermal transfer printing machine at 160°C, 180°C and 200°C temperatures. In this process, printing pressure and printing time were kept constant.

2.4. Measurement of Printability Properties

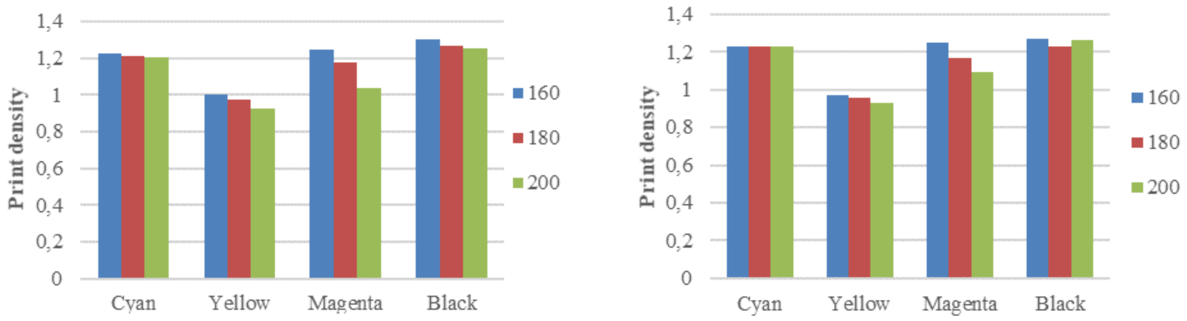
CIE L*, a*, and b* values of the printed uncoated samples and the printed samples coated with EA oligomer were measured by the D50 illuminant/2° observer values using X-Rite eXact Densitometer [9]. BYK Portable glossmeter (BYK-Gardner Gmbh, Geretsried, Germany) was used based on ISO 2813 (2014) to determine the gloss values of the printed samples. Both CIE L*, a*, and b* color values and gloss values of the printed samples were measured immediately after printed and the washing. Also, Resistance of printed fabrics to fade was measured according to BS 4321 using Solorbox 1500 device.

2.5. Washing Conditions

The obtained printed, bamboo jersey fabrics were washed once by using ECE non-ionic detergent (4 g/L) solution in water at 40ºC for ½ h [10]. The quality of the printing process was evaluated both before and after the washing step.

III. RESULTS AND DISCUSSIONS

Figure 1 shows the print density values of Cyan, Magenta, Yellow and Black colors printed on bamboo jersey fabrics at different temperature changes and after washing process. It saw that there is a decrease in the density values depending on the increase in temperature values in all colors. After the washing process, the density values of the colors were not changed negatively.

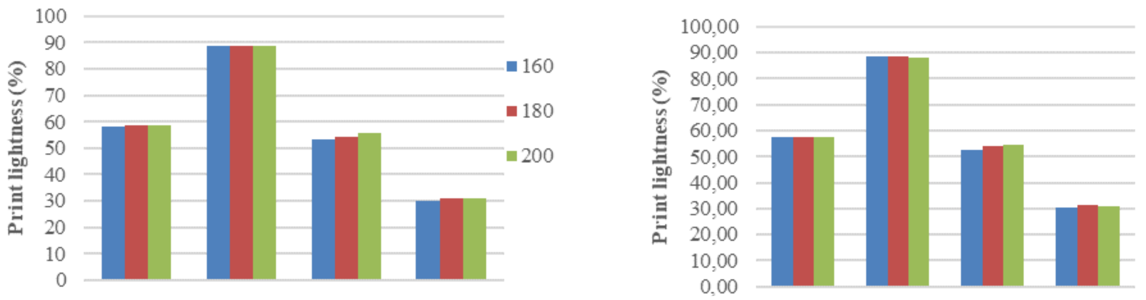


Print color before washing

Print color after washing

Figure 1. The print density values before and after washing

Figure 2 shows the print lightness values of Cyan, Magenta, Yellow and Black colors printed on bamboo jersey fabrics at different temperature changes and after washing process. It is understood from the obtained data that neither the change of the temperature values nor the washing process has any effect on the printing lightness values.

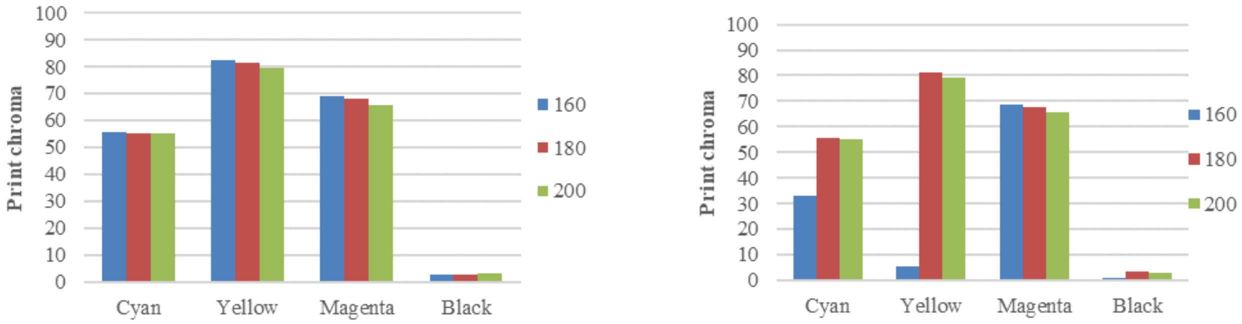


Print color before washing

Print color after washing

Figure 2. The print lightness values before and after the washing

Figure 3 shows the print chroma values of Cyan, Magenta, Yellow and Black colors printed on bamboo jersey fabrics at different temperature changes and after washing process. Depending on the increase in temperature, there is a slight decrease in the print chroma values of the print colors. This decrease was slightly increased after washing.



Print color before washing

Print color after washing

Figure 3. The print chroma values before and after the washing

Table 1. The gloss values before and after washing of bamboo jersey fabrics

Bamboo jersey fabrics	Gloss before washing	Gloss after washing
	2,3	2,6

Bamboo jersey fabrics before and after washing preserved their gloss values (Table 1).

Table 2. The print gloss values before the washing

Print Gloss before washing	160 °C	180 °C	200 °C
Cyan	1,6	2,2	2,1
Yellow	3,0	3,3	3,3
Magenta	2,0	2,1	2,0
Black	1,7	1,80	1,8

In Table 2, it is seen that the gloss values of Cyan, Magenta and Black colors after printing are slightly lower than the gloss of unprinted Bamboo jersey fabrics. However, the change in the temperature values did not significantly affect the print gloss value.

Table 3. The print gloss values after the washing

Print Gloss after washing	160 °C	180 °C	200 °C
Cyan	0,8	0,8	0,9
Yellow	2,1	1,9	2,1
Magenta	1,0	0,9	0,8
Black	0,6	0,7	0,6

Printed gloss values decreased after washing. The change in the temperature values did not affect the print gloss value (Table 3).

Light fastness measurement was made according to BS ISO 12040: 1997 [11]. According to this standard, it means 1 very little, 2 little, 3 medium, 4 fairly good, 5 good, 6 very good, 7 excellent and 8 spectacular.

Table 4. The evaluation of light fastness of printed fabrics

Light fastness before washing	160 °C	180 °C	200 °C
Cyan	7	6	5
Magenta	7	6	5
Sarı	7	6	5
Siyah	7	6	5

Table 4 shows the results of evaluating the light fastness of the printed fabrics according to the blue scale before the washing. It is seen that the light fastness values of the colors decrease due to the temperature increase in the table. The best fastness value was obtained at 160 ° C.

IV. CONCLUSIONS

The fact that the Cyan, Magenta, Yellow and Black colors printed on the Bamboo jersey fabrics do not change in the different print temperature values and the printing density after washing show that the consistency in the printing density value is maintained.

Both the change in print temperature values and the print lightness values of the wash process did not affect.

The print chroma values do not change significantly due to different print temperature values and washings. It has shown that the color universe, which is can be obtained after print, preserves consistency.

Before and after washing Bamboo jersey fabrics retained their gloss values. The value of the print gloss was not significantly influenced by the change in temperature values. But, the print gloss values were noticeably reduced after washing.

Depending on the print temperature increase reduced in light fastness of print colors. The best light fastness value was obtained at 160 ° C.

In general, it is concluded results that Bamboo jersey fabrics have a printable structure in thermal transfer printing and preserve the consistency of the print outside the print gloss after the washing process. While the increase in the print temperature value does not significantly affect the print quality, it affects the light fastness of the print.

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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

Utilizing Recycled High Density Polyethylene Caps of Polyethylene Terephthalate Bottles in Composite Plates

Kompozit Plakalarda Polietilen Tereftalat Şişelerinin Geri Dönüştürülmüş Yüksek Yoğunluklu Polietilen Kapaklarının Kullanımı

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Abstract

In this study, composite plates that are consisted of recycled high density polyethylene (HDPE) polymer matrix and reinforced with woven fabrics (cellulosic fiber content; flax and jute) are manufactured in order to contribute polymer waste management. Used HDPE bottle caps are collected for recycling and hot press method is used to produce HDPE matrix plates. Plates are than reinforced with 100% jute and 100% flax woven fabrics with different fabric sequence combinations by hot press machine. Temperature, production time, pressure and cooling rate are the crucial parameters that are optimized for forming composite materials. These bi-layered and three-layered composite plates are physically and mechanically tested for appropriate end use areas.

Keywords: Composite, recycling, packaging waste, textile

Öz

Bu çalışmada, polimer atık yönetimine katkıda bulunmak amacıyla geri dönüştürülmüş yüksek yoğunluklu polietilen (HDPE) polimer matrisinden oluşan ve dokuma kumaşlarla takviye edilmiş (selülozik elyaf içeriği; keten ve jüt) kompozit plakalar üretilmiştir. Kullanılmış HDPE şişe kapakları geri dönüşüm için toplanmış ve HDPE matris plakaları üretmek için sıcak presleme yöntemi kullanılmıştır. Plakalar, sıcak pres makinesi ile farklı kumaş sıralımlarına sahip %100 jüt ve %100 keten dokuma kumaşlarla takviyelendirilmiştir. Sıcaklık, üretim süresi, basınç ve soğutma oranı, kompozit malzemeleri oluşturmak için optimize edilmiş kritik parametrelerdir. Bu çalışma kapsamında üretilmiş olan iki katmanlı ve üç katmanlı kompozit plakalar, uygun son kullanım alanları için fiziksel ve mekanik olarak test edilmiştir.

Anahtar Kelimeler: Kompozit, geri dönüşüm, ambalaj atığı, tekstil

I. INTRODUCTION

Composite structures are the materials composed of at least two types of materials by physical bonding [1]. They are generated in an attempt to obtain outstanding performance features and matchless mechanical properties with constitution of different materials that have especial internal and external structure [2]. It is aimed to improve many properties by combining the best properties of different materials such as thermal conductance, fracture toughness, fatigue strength, corrosion resistance, abrasive strength and aesthetic appearance [3]. In composite materials, mostly fibers are used as reinforcement materials [4]. Fibers are the critical elements of composite structures, as they satisfy the favored situations and transfer strength to the matrix material. The overall performance of a fiber composite depends on fiber's form, orientation, composition and the mechanical properties of the matrix material [5].

The usage of natural fibers as renewable resources in composite industry have increased due to increased awareness of people [6]. Flax and jute are the most preferred natural fibers that are used as reinforcement materials. Flax fiber has

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low specific weight, low energy consumption during processing and relatively low cost. It is stated in the literature that, flax fiber reinforced composites exhibit good mechanical properties due to the distinctive surface morphology of the flax. However, the incompatibility of flax fiber with polymeric resins results in poor fiber/matrix interfacial bonding strength [7]. Jute fiber and its composites are non-abrasive, porous, good insulator and combustible materials. It is a promising reinforcement material due to its low cost, low density, high specific strength and modulus, easy availability and renewability. The drawback of the jute fiber is its high moisture absorption [8].

Packaging is surrounding materials, which is made for protecting products from the environment and also preserves the products' quality, thereby reducing damaged products and waste [9]. There are 5 main types of packaging which are metal, glass, cardboard, carton and plastics and these can be found in the market in several types and forms. Plastics are the most used material for packaging goods due to their lightweight barrier properties and flexibility [10]. In the last 5 years, 3 million tons of plastic materials are used in Turkey just for packaging sector and 37% of it belongs to polyethylene (both high density polyethylene and low density polyethylene) packaging [11]. It is one of the most widely used thermoplastic material owing to its toughness, low moisture absorption, excellent chemical inertness, low coefficient of friction, ease of processing and low electrical conductivity [12].

High density polyethylene (HDPE) is a strong, high density, moderately stiff plastic with a highly crystalline structure. It is frequently used in milk jars, laundry detergent bottles, garbage bins, and cutting boards [12]. High density polyethylene (HDPE) is one of the most common polyethylene type especially used in polyethylene terephthalate (PET) bottle caps which can be reusable after melting and re-shaping processes owing to its thermoplastic characteristics. When its huge percentage in solid wastes and ease of accessibility are taken into consideration, recycling of HDPE package wastes becomes a very important issue in environmental aspects.

Recycling has been a significant matter in last decades, related to increasing environmental pollution. There is an increasing tendency among state and local governments about recycling action [13]. Recycling includes reusing the materials when their life cycle is ended, by regaining these materials in production of new goods [14].

In this context, in this study HDPE bottle caps in the waste state were collected and reinforced with jute and flax woven fabrics and aimed to contribute to waste management as well as making an innovative material design.

II. EXPERIMENTAL SET-UP AND PROCEDURE

2.1. Materials

In this study, high density polyethylene (HDPE) bottle caps having density of 0.74 g/cm^3 are used as matrix material whereas flax and jute woven fabrics are used as reinforcement materials.

Properties of reinforcement materials are given in Table 1.

Table 1. Physical properties of reinforcement materials

Sample Code	Fiber content	Weave Type	Yarn Count	Areal Density (g/m^2)	Fabric Count (e.p.c x p.p.c)
F	Flax	Basket (2x1)	Ne 3.2 (weft) Ne 5.9 (warp)	294	16 x 9
J	Jute	Plain	Ne 2.8 (weft) Ne 2.6 (warp)	241	7 x 6

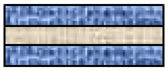
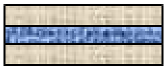
*e.p.c.: ends per cm, p.p.c: picks per cm

2.2. Composite Production

For composite production, hot press machine is used. Several preliminary productions are performed to determine the most suitable form of matrix and reinforcement material and optimum production parameters. Based on the experience of preliminary experiments, recycled HDPE matrix plates are pre-produced by hot press machine. 36 plastic water bottle caps made of HDPE are placed in a 6 x 6 matrix to ensure homogeneous distribution for larger plate productions. They are covered with Teflon sheet to prevent contact between machine and polymer materials before starting hot press operation. Optimum production parameters are performed (160 °C, 20 tons, 1.5 hours with pressure + 3 hours without pressure). The compressed HDPE plate is slowly cooled under decreasing pressure for 3 hours in order to get a homogenous and smooth matrix plate. The pre-produced matrix plates and fabric reinforcement material (jute and flax) are covered by Teflon paper together and place into the hot plate machine. The same optimized production parameters are applied to the composites.

Different composite designs are prepared by changing fabric and matrix plate layout. The details of these composite designs are listed in Table 2.

Table 2. Sample codes

Specimen Code	Layers	Components
HF		Recycled HDPE Plate Flax Fabric
HFH		Recycled HDPE plate Flax Fabric Recycled HDPE Plate
FHF		Flax Fabric Recycled HDPE Plate Flax Fabric
HJ		Recycled HDPE Plate Jute Fabric
HJH		Recycled HDPE Plate Jute Fabric Recycled HDPE Plate
JHJ		Jute Fabric Recycled HDPE Plate Jute Fabric

The pictorial views of the composite designs are given in Figure 1 and Figure 2.

Figure 1. H-F-H, H-F and F-H-F composites (left to right)

Figure 2. H-J-H, J-H-J and H-J composites (left to right)

III. ANALYSES

Thicknesses of composite materials are measured by digital thickness gauge. The densities of the composite structures are calculated from the measured average weight and calculated volume data.

Composites are tested by their water absorption and tensile properties. In water absorption test, both composite and fabric thicknesses are measured and surface areas are calculated. After their dry weights are measured by using precision scale, samples are immersed in water one by one and their wet weights are measured. Immersion period is 30 seconds. Results of this experiment are given as % water absorption of produced composite.

Tensile test is conducted by using Titan Tensile Tester according to TS EN ISO 13934-1 standard.

IV. RESULTS AND DISCUSSIONS

4.1. Thickness

Thicknesses of HDPE matrices are measured as 0.30 mm whereas thickness of flax and jute fabrics are 0.61 and 0.58 mm, respectively. When composite structures are taken into consideration, it can be said that designs including 1 matrix and 1 fabric layer (HF and HJ) have very similar thicknesses (0.51 and 0.63 mm, respectively) with fabric thicknesses. On the other hand, especially designs including 1 matrix plate in the middle of the 2 fabrics have the maximum thicknesses (0.87 mm for FHF and 1.02 mm for JHJ).

4.2. Density

Densities of the samples are listed in Table 3. According to the calculated data, it can be said that due to the air gaps in the structure, fabrics have less densities than matrix plates. Especially for jute fabric reinforced composites, the increment in the amount of matrix plate increase the density while the increase in jute fabric layers decrease the density of the composite structure. For flax fabric reinforced composites, it is not possible to say such clear comments which can be resulted from unwanted air gaps regarding to un-wetted fibers in the structure.

Table 3. Densities of samples

Sample Codes	Average Weight (g)	Volume (cm^3)	Density (g/cm^3)
H	3.40	4.57	0.74
HF	8.19	7.74	1.06
HFH	9.72	9.71	1.00
FHF	11.71	13.13	0,89
HJ	6.71	9.51	0.71
HJH	9.13	10.38	0.88
JHJ	10.57	15.29	0.69

4.3. Water absorption

Water absorption test results are given in Table 4. According to the data calculated, water absorbency percentages of HDPE plates are zero, as expected based on the hydrophobic characteristics of polymers. Similarly, composite designs composed of 2 matrix plates in both sides of core fabric have almost 0% water absorbency which means that the reinforcement fabric is covered by HDPE matrices as well. On the other hand, owing to hydrophilic characteristics of natural fabrics, in designs composed of two fabrics in both sides and core matrix in the middle have higher water absorbency percentages which means the inner matrix plate does not have capability to wet all the outer fabric layers to make them waterproof.

Table 4. Water absorbency test results

Samples Codes	Dry Weight (g)	Wet Weight (g)	Water Absorbency (%)
H	0.53	0.53	0.00
HF	0.95	1.08	13.68
HFH	1.43	1.43	0.00
FHF	1.47	2.28	55.10
HJ	0.98	1.02	4.08
HJH	1.63	1.63	0.00
JHJ	1.56	1.92	23.08
J	0.63	1.62	157.14
F	0.7	1.69	141.43

4.4. Tensile Test

Tensile test results are given in Table 5. According to the results, tensile strength of jute fabric is lower than flax fabric. In all two layer designs (HF; HJ), tensile strength increases with reinforcing fabrics in comparison to both single fabrics and matrix plates. Moreover, when three layered designs are compared with each other, all samples including matrix side layers (HFH; HJH) have higher tensile strength values than fabric side layers (FHF; JHJ). This can be explained by poor wetting of the core matrix. It is known that our matrix plates are softened under pressure and heat but not be as liquid as water therefore its wetting ability is limited. With the designs including two matrix plates in side layers together with fabric core had better wetting ability, which results in better fiber to matrix interactions leading to improved mechanical properties.

Table 5. Tensile strength test results

Sample Codes	Tensile Strength (MPa) ± SD	
	Warp Direction	Weft Direction
H	18.05±1.25	
HF	53.76±0.80	25.05±0.82
HFH	54.78±3.50	54.97±1.66
FHF	47.48±1.26	54.20±0.76
HJ	21.22±1.30	13.72±0.07
HJH	28.74±3.25	23.70±10.47
JHJ	16.25±0.39	10.52±0.40
J	17.54±0.70	16.95±1.10
F	26.19± 0.24	33.23±0.19

V. Conclusions

Packaging waste constitutes the main part of the solid waste problem around the world. HDPE, used in PET bottle caps, becomes municipal solid waste that accumulates in nature eventually. Thus, composites designed in this study are mostly focused on reducing packaging waste by recycling the bottle cap waste and regaining of used HDPE polymer. Results of experiments show that appropriate designs can be selected for the end use application in which moderate tensile strength and good waterproof properties are required. Mechanical properties of the composite panels can be improved with additional chemicals for future works.

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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

One Bath Dyeing of PBT/Nylon Blended Seamless Fabrics

PBT/Naylon Karışımlı Seamless Kumaşların Bir Banyoda Boyanması

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Abstract

Polyesters (PES) fibers are one of the most important polymers for textile industry due to their large production amounts. Poly (butylene terephthalat) (PBT) fibers are important aromatic polyesters known as their good elasticity and easy dyeing properties. PBT is manufactured by again polycondensation reaction with BDO (1,4-butanediol) and TPA or DMT, can be easily dyed at low temperatures with disperse dyes without carrier and is known as its very good elasticity properties. Generally PBT fibers are used in intimate, ready-to-wear, active and sportswear apparels, swimwear, carpets, automotive and home upholstery applications. Seamless technology is capable to meet the needs of consumers with performance, functionality and comfort. This technology has some advantages over the cutting and sewing. Seamless technology improves the aesthetic value and comfort, fredom of body movement, gives softness to the garment and minimizes the surface friction of the seams. In seamless machines, gloves, hats, socks, sweaters, sportswear can be produced. PBT fibers can be used in seamless sport wears, tights and intimates. These fibers provide more elasticity, comfort and softness on seamless sportswear than polyester fibers due to their good elasticity properties. In seamless technology polyester fibers generally are used with other fibers such as nylon, elastane.

In this study, PBT/Nylon blend knitted seamless fabrics were dyed with disperse, acid and metal complex dyes at 98ºC in one bath. The color strength, colorimetric properties and fastness properties (rub and wash fastness) of these fabrics are investigated and compared. Moreover, the comparison of water comsumptions and costs of one and two baths dyeings are given on this study.

Keywords: Poly (buthylene terephthalate) fiber, PBT, elastic polyesters, easy dyeing, elasticity, seamless technology

Öz

Polyester (PES) elyafı, yüksek üretim miktarları nedeniyle tekstil endüstrisi için en önemli polimerlerden biridir. Poli (butilen terephthalat) (PBT) lifleri, iyi esneklikleri ve kolay boyama özellikleri olarak bilinen önemli aromatik polyesterlerdir. PBT, BDO (1,4 bütandiol) ile TPA veya DMT'nin polikondensasyon reaksiyonuyla üretilir, keriyer olmadan dispers boyarmaddelerle düşük sıcaklıklarda kolayca boyanabilir ve çok iyi esneklik özellikleri olarak bilinir. Genel olarak PBT elyafları, iç çamaşırı, hazır giyim, aktif ve spor giyim eşyalarında, mayolarda, halılarda, otomotiv ve ev döşemelerinde kullanılır. Seamless teknolojisi, performans, işlevsellik ve konfor ile tüketicilerin ihtiyaçlarını karşılayabilir. Bu teknoloji, kesim ve dikme ürünlerinde bazı avantajlara sahiptir. Kusursuz teknoloji, estetik değeri ve konforu artırır, vücudun daha özgür hareket etmesine yardımcı olur, giysiye yumuşaklık verir ve dikişlerin yüzey sürtünmesini en aza indirir. Dikişsiz makinelerde eldiven, şapka, çorap, kazak, spor giyim üretilebilir. PBT elyafları dikişsiz spor giyim, tayt ve iç çamaşırlarında kullanılabilir. Bu elyaflar, iyi esneklik özellikleri nedeniyle dikişsiz spor giyimde polyester elyaflardan daha fazla esneklik, rahatlık ve yumuşaklık sağlar. Dikişsiz teknolojide polyester elyaflar genellikle naylon, elastan gibi başka elyaflarla birlikte kullanılır.

Bu çalışmada PBT/Naylon karışımı örme seamless kumaşlar tek bir banyoda 98ºC'de dispers, asit ve metal kompleks boyarmaddelerle boyanmıştır. Bu kumaşların renk değerleri, kolorimetrik özellikleri ve haslık özellikleri (sürtme ve yıkama haslığı) araştırılmış ve karşılaştırılmıştır. Ayrıca, bu çalışmada tek banyolu ve çift banyolu boyamalarda harcanan su tüketimleri ve maliyetler karşılaştırılmıştır.

Anahtar Kelimeler: Poli(bütilen tereftalat) lifi, PBT, elastik polyesterler, kolay boyama, elastikiyet, seamless technolojisi

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I. INTRODUCTION

As the population increases natural fibers have failed to satify needs of humankind, and thus synthetic fibers were started to produce [1, 2]. Polyethylene terephthalate (PET) polyester, which was discovered by Whinfield and Dickson, was first produced commercially in 1941 [3]. The polyester is obtained by extracting the polyethylene terephthalate (PET) polymer formed by polymerization of ethylene glycol and ethylene. Polyesters (PES) are known as the most important fiber polymers for the textile industry [4, 5]. Polyester fibers are attractive for using in medicine, clothing, sports and various industrial fields due to their economic performance. When the polyester fibers are modified physically and chemically, high performance fibers are obtained [6]. Poly (butylene terephthalate) (PBT) was produced by Carothers and Hill (DuPont). PBT is a semi-crystalline polymer with similar properties to PET in both color and colorless properties and known as 4 GT or polytetramethylene terephthalate (Fig. 1). PBT is obtained by polycondensation reaction of purified terephthalic acid (PTA) or dimethyl terephthalate (DMT) with 1,4-butanediol (BDO) [4, 7].

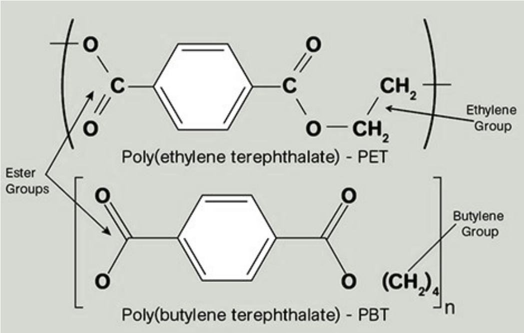


Figure 1. Chemical Structures for PET and PBT Repeating Units [8]

PBT fibers have very low glass transition point (Tg) values, so they can easily be dyed at atmospheric conditions without carrier. Commercial PBT fibers are produced by Zimmer and Ticona and known as Celanex® [4].

Nylon was discovered by Wallace Carothers in DuPont in 1928. Nylon (polyamide, PA) is a synthetic polymer called polyamide consisting of monomers of amides in the backbone chain and is widely used in the textile industry as PA 6.6 (nylon 6.6) and PA 6 (nylon 6). PA 6.6 is obtained by polymerizing adipic acid $[HOOC - (CH_2)_4\text{-}COOH]$ with hexamethylene diamine $[H_2N - (CH_2)_6\text{-}NH_2]$ (Fig. 2) [9, 10].

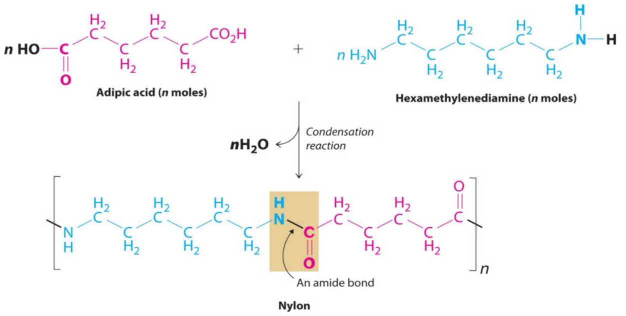


Figure 2. Chemical structure of nylon [11]

Nylon 6,6, is one of the most common synthetic fibers, has extensive use in many areas thanks to its excellent physical properties, good durability [12]. PBT and nylon fibers show similarities in many respects. Both polymers have fast crystallization ability, good mechanical properties, good solvent strength and similar melting temperatures (table 1) [5].

Table 1. Physical properties of PBT and nylon (PA 6 and PA6.6) fibers [5, 13]

Physical Properties	PBT	PA6.6	PA6
Melting Point (˚C)	224	265	220
Glass Transition Temperature (Tg˚C)	20-40	50-90	40-80
Resistance	+	++	++
Chromaticity	+(+)	++	++
Dimensional Stability / Pulling	(+)	+(+)	+(+)
Elasticity, Dry	++	+	+
Chloride resistance	++	+	+
Resistance to hot alkalis	-	++	+

++ good / strong; + moderate; – bad / weak

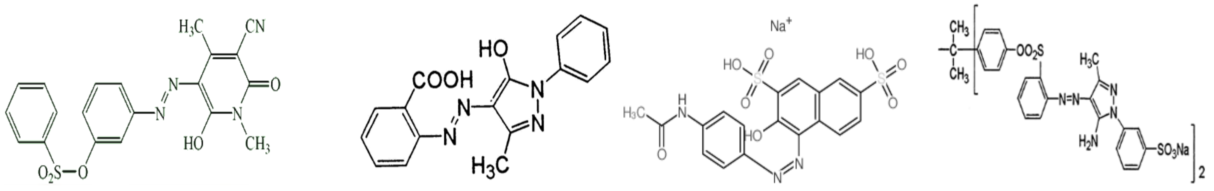
In recent years, many researchers have studied dyeing conditions for PBT and blended yarns with PBT [14-25]. In this study, PBT/Nylon (PBT/PA) blended seamless fabrics were dyed with disperse, acid and metal complex dyes at 98˚C. The color strength, colorimetric properties and fastness properties (rub and wash fastness) and differences of these fabrics were examined and compared.

II. EXPERIMENTAL SET-UP AND PROCEDURE

75% Naylon-25%PBT seamless knitted farbics were used in this study. All fabric samples were dyed in one bath and rinsed after dyeing. Dyeing processes were carried out using commercially available low, medium and high energy disperse dyes (table 1), acid and metal complex dyes. Four different color shades are used for this study such as yellow, green, red and salmon. Disperse dyes and acid dyes used in one bath dyeing were given in table 2 and figure 3.

Table 2: Structures and C.I. numbers of disperse and acid and metal complex dyes

Disperse dyes	**Molecular weight of disperse dyes**	**Acid and metal complex dyes**
Yellow P-6G (C.I. Dispers Yellow 114)	High energy	Red SRL (C.I. Acid Red 414)
Orange S2PR	High energy	Yellow KGLN (C.I. Acid Yellow 59)
Yellow SE-2GR	Medium energy	Yellow M4GL (C.I. Acid Yellow 79)
Blue SE-5R	Medium energy	
Blue ENF	Low energy	
Yellow ENF	Low energy	
Red ENF	Low energy	



C.I. Dispers Yellow 114 [26] C.I. Acid yellow 59 [27] C.I. Acid Red 414 [28] C.I. Acid Yellow 79 [29]

Figure 3: Chemical structures of disperse, acid and metal komplex dyes used in dyeing

Dyeing processes of PBT and Nylon blended fabrics were carried out in Laboratory Eco Dyer at 1:10 liquid ratio at 98 °C. The fabrics rinsed with warm water and tumbler dried.

III. ANALYSIS

The *K/S* values were measured with using Spectrophotometer under illuminant D65, using 10° Standard observer for each dyed samples. The colour strength value K/S is calculated by using the Kubelka-Munk equation. The equation of *K/S, Eqation (1)* is given at below:

$K/S = (1-R^2)/2R$ (1)

The wash and rub fastness properties of dyed fabrics were investigated. The wash fastness test was performed according to ISO 105:C06 B2S test. Both dry and wet rub fastness tests were performed according to ISO 105: X12 protocol. Overall fastness properties were evaluated by using ISO grey scales in the light box. The energy and water savings obtained by dyeing the fabrics in one bath instead of two baths were also calculated.

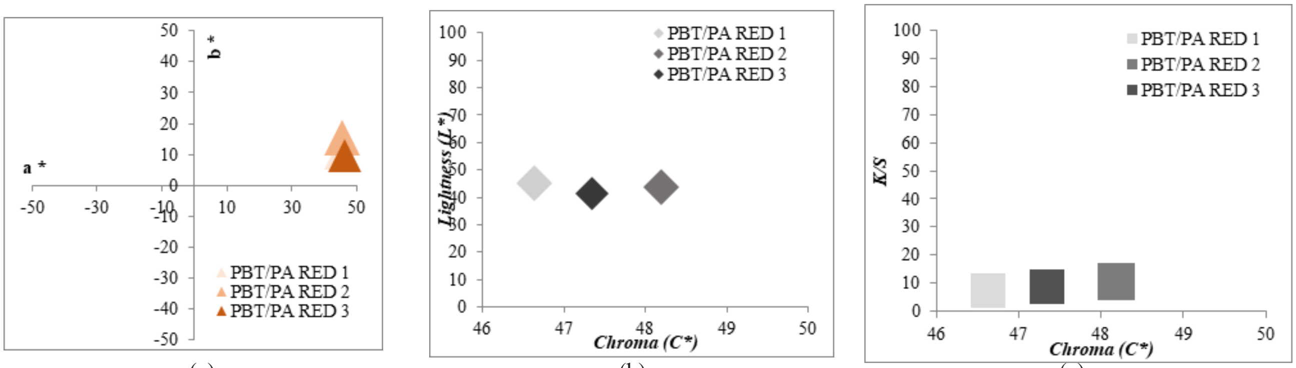
IV. RESULTS AND DISCUSSIONS

The colorimetric properties of red dyed PBT/Nylon (PBT/PA) blended fabrics are given on Table 3 and Figure 4 (a)-(c). With these experiments, color diffrerences between dyed fabrics with only metal complex dyes and both metal complex and disperse dyes were investigated.

Table 3: Colorimetric properties of PBT/PA blended fabrics dyed with only metal complex dyes and both metal complex and disperse dyes.

Samples	Dyes		Molecular weight of disperse dyes	Color	*K/S*	*L**	*a**	*b**	*C**	*h*°	
	Metal complex										
PBT/PA RED 1	Red SRL		-	Red	7,15	45,2	45,3	10,9	46,6	13,5	🟥
	Metal complex	**Disperse dyes**									
PBT/PA RED 2	Red SRL	Red ENF	Low energy	Red	10,3	43,7	45,6	15,5	48,1	18,7	🟥
	Metal complex	**Disperse dyes**									
PBT/PA RED 3	Red SRL	YellowENF	Low energy	Red	8,45	41,6	46,4	9,4	47,3	11,4	🟥

Color strength values of red dyed PBT/PA blended fabrics change range from 7,15 to 10,3. The highest color strength value is observed on PBT/PA RED 2 sample. This fabric was dyed with metal complex dye and low energy red disperse dye. PBT/PA fabric dyed with only metal complex red dyed showed lowest color strength value. PBT/PA blended fabrics dyed with metal complex and disperse dyes in one bath exhibited better color strength values than those of fabrics dyed with only metal complex dyes. Therefore, dyeing with metal complex and disperse dyes in one bath could be recommended.



(a) (b) (c)

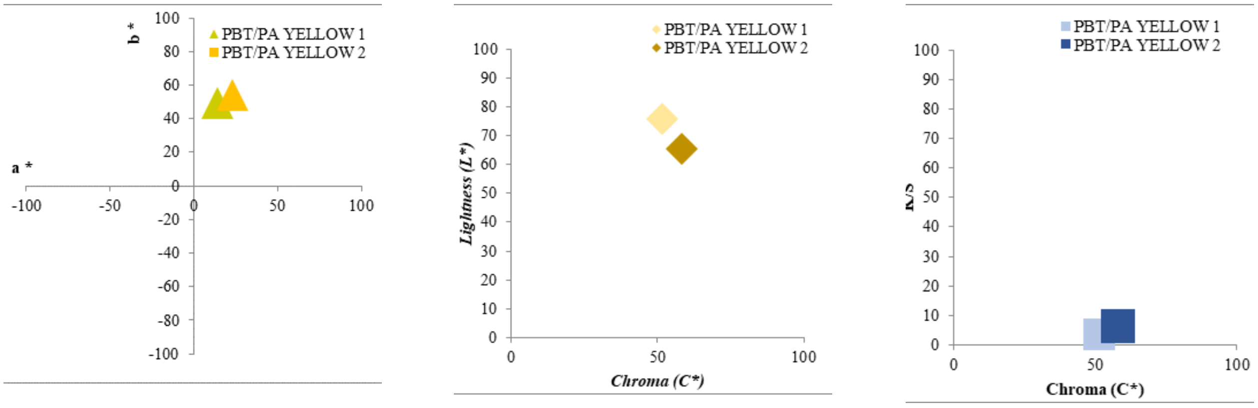
Figure 4: graphics of dyed PBT/PA blended fabrics; (a) redness (a^*) – yellowness (b^*) plots, (b) Lightness (L^*)-Chroma ($^*C$) plots, (c) color strength (*K/S*) – Chroma ($^*C$) plots

As seen on Figure 4, PBT/PA fabric dyed with only metal complex dyes exhibited similar redness-yellowness values. But its chroma values are lower than that of PBT/PA blended fabrics dyed with both metal complex and disperse dyes. The colorimetric properties of yellow dyed PBT/Nylon blended fabrics are given on Table 4 and Figure 5 (a)-(c).

Table 4: Colorimetric properties of PBT/PA blended fabrics dyed wit only disperse dyes and both metal complex and disperse dyes.

Samples	**Dyes**		**Molecular weight of disperse dyes**	**Color**	***K/S***	L^*	a^*	b^*	C^*	h^o	
PBT/PA YELLOW 1	**Disperse dyes**		High energy	Yellow	3,30	75,8	13,9	49, 5	51,4	74,2	
	Yellow P6G	Oranj S2PR									
PBT/PA YELLOW 2	**Metal complex**	**Disperse dyes**	Low energy	Yellow	6,20	65,5	23,1	53,5	58,34	66,5	
	Yell. KGLN	Red ENF									

Dyed samples displayed orange-yellow color shades. Samples dyed with both metal complex and disperse dyes displayed darker than that of samples dyed with only disperse dyes. Color strength values of yellow dyed PBT/PA blended fabrics change range from 3,3 to 6,2. The highest color strength value is observed on PBT/PA YELLOW 2 sample which was dyed with metal complex dye and low energy class red disperse dye. Only dipserse dyed PBT/PA fabric showed lowest color strength value. Dyed PBT/PA blended fabrics with metal complex and disperse dyes in one bath exhibited better color strength values than those of fabrics dyed with only disperse dyes. Therefore, dyeing with metal complex and disperse dyes in one bath could be recommended.

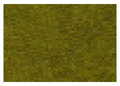
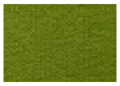


(a) (b) (c)

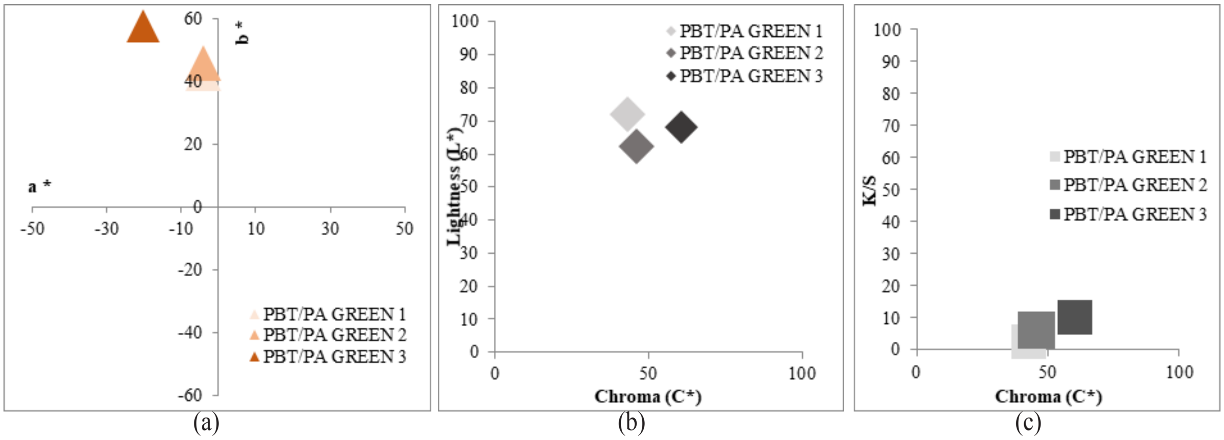
Figure 5: graphics of dyed PBT/PA blended fabrics; (a) redness (*a**) – yellowness (*b**) plots, (b) Lightness (*L**)-Chroma *(*C*) plots, (c) color strength (*K/S*) – Chroma *(*C*) plots

As seen on figure 5, PBT/PA fabric dyed with only disperse dyes exhibited similar redness-yellowness values. But its chroma values are lower than that of PBT/PA blended fabrics dyed with both metal complex and disperse dyes. The colorimetric properties of green dyed PBT/Nylon blended fabrics are given on table 6 and Figure 6 (a)-(c).

Table 6: Colorimetric properties of PBT/PA blended fabrics dyed with only disperse dyes, both metal complex and disperse dyes and both acid and disperse dyes.

Samples	Dyes		Molecular weight of disperse dyes	Color	*K/S*	*L**	*a**	*b**	*C**	*h*°	
PBT/PA GREEN 1	**Disperse dyes**		Medium energy	Green	2,5	71,9	-4,04	42,84	43,1	95,4	
	Yellow SE-2GR	Blue SE-5R									
PBT/PA GREEN 2	**Metal complex**	**Disperse dyes**	Low energy	Green	5,8	62,3	-4,2	45,8	45,9	95,2	
	Yell. KGLN	Blue ENF									
PBT/PA GREEN 3	**Acid Dyes**	**Disperse dyes**	Low energy	Green	10,1	68,2	-19,8	57,4	60,7	109,1	
	Yell M4GL	Blue ENF									

Dyed samples displayed green color shades. Color strength values of green dyed PBT/PA blended fabrics change range from 2,5 to 10,1. The highest color strength value is observed on PBT/PA YELLOW 2 sample which was dyed with Acid dye and low energy class red disperse dye. PBT/PA fabrics dyed with only disperse dyes showed lowest color strength value. PBT/PA blended fabrics dyed with metal complex, acid and disperse dyes in one bath exhibited better color strength values than those of fabrics dyed with only disperse dyes. Therefore, dyeing with acid and disperse dyes in one bath could be recommended.



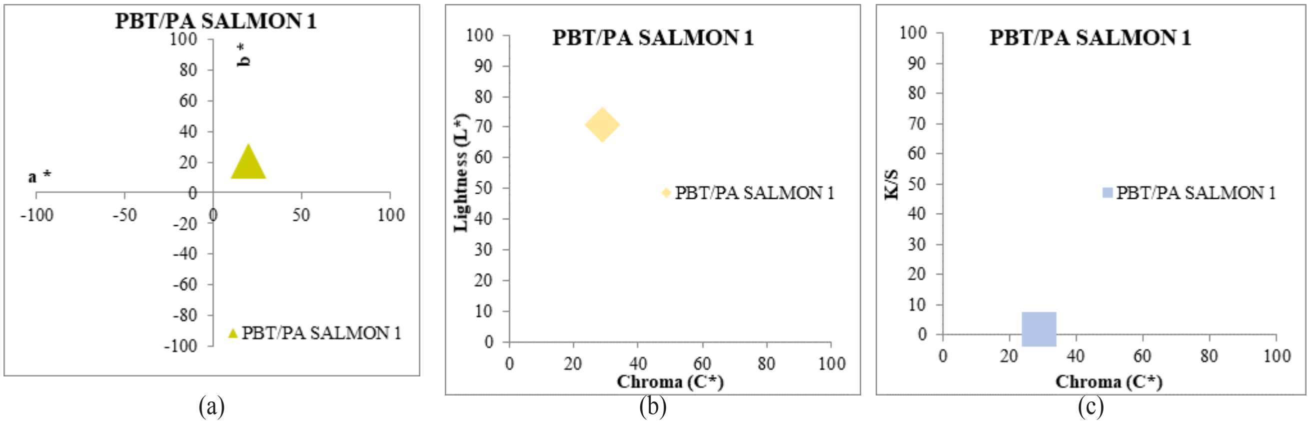
(a) (b) (c)

Figure 6: graphics of dyed PBT/PA blended fabrics; (a) redness (*a**) – yellowness (*b**) plots, (b) Lightness (*L**)-Chroma *(*C)* plots, (c) color strength (*K/S*) – Chroma *(*C)* plots

As seen on figure 6, PBT/PA fabric dyed with only disperse dyes exhibited similar redness-yellowness values as PBT/PA fabric dyed with metal complex and disperse dyes. PBT/PA fabrics dyed with acid and disperse dyes displayed highest color strength and redness-yellowness values. The colorimetric properties of salmon dyed PBT/Nylon blended fabrics are given on table 7 and Figure 7 (a)-(c).

Table 7: Colorimetric properties of PBT/PA blended fabrics dyed with only low energy disperse dyes

Samples	**Dyes**			**Molecular weight of disperse dyes**	**Color**	***K/S***	***L****	***a****	***b****	***C****	***h°***	
	Disperse dyes											
PBT/PA SALMON 1	Red ENF	Yellow ENF	Blue ENF	Low energy	Salmon	1,5	70,7	19,8	21,2	29,1	47	



(a) (b) (c)

Figure 7: graphics of dyed PBT/PA blended fabrics; (a) redness (*a**) – yellowness (*b**) plots, (b) Lightness (*L**)-Chroma *(*C)* plots, (c) color strength (*K/S*) – Chroma *(*C)* plots

Dyed sample displayed salmon color shade. Color strength value of salmon color dyed PBT/PA blended fabric is 1,5. Wash and rub fastness properties of dyed samples were analyzed. Rub and wash fastness levels of the PBT/PA blendend fabrics were given on table 8.

Table 8: Rub and wash fastness properties of all dyed samples

Samples	Dyes		Molecular weight of disperse dyes	K/S	Rub fastness		Wash Fastness					
					Wet	Dry	WO	PC	PES	N6,6	CO	AC
PBT/PA RED 1	**Metal complex** Red SRL – 1		-	7,15	5	5	4/5	4/5	4/5	4	4/5	4/5
PBT/PA RED 2	**Metal complex** Red SRL – 1	**Disperse dyes** Red ENF	Low energy	10,3	5	5	4/5	5	4/5	3/4	4/5	4
PBT/PA RED 3	**Metal complex** Red SRL – 1	**Disperse dyes** YellowENF	Low energy	8,45	5	5	4/5	5	5	4	4/5	5
PBT/PA YELLOW 1	**Disperse dyes** Yellow P6G	Oranj S2PR	High energy	3,30	5	5	4/5	5	4/5	4	4/5	4
PBT/PA YELLOW 2	**Metal complex** Yell. KGLN	**Disperse dyes** Red ENF	Low energy	6,20	5	5	4/5	5	4/5	3/4	4/5	4
PBT/PA GREEN 1	**Disperse dyes** Yellow SE-2GR	Blue SE-5R	Medium energy	2,5	5	5	5	5	5	4/5	5	4/5
PBT/PA GREEN 2	**Metal complex** Yell. KGLN	**Disperse dyes** Blue ENF	Low energy	5,8	5	5	5	5	5	3/4	4/5	4/5
PBT/PA GREEN 3	**Acid Dyes** Yell M4GL	**Disperse dyes** Blue ENF	Low energy	10,1	5	5	5	5	5	4	5	4/5
PBT/PA SALMON 1	**Disperse dyes** Red ENF Blue ENF	Yellow ENF	Low energy	1,5	5	5	5	5	4/5	3/4	5	4

All dyed samples exhibited excellent wet and dry rub fastness properties. Wash fastness values are generally exhibited good to excellent wash fastness by 5 grey scale rating for staining except nylon 6.6 and acetate fabrics. As known, metal complex and acid dyes have an interest in nylon fibers. Therefore, nylon fabrics in adjacent fabrics exhibited 3/4 – 4/5 staining values. PBT/nylon fabrics dyed by high energy disperse dyes exhibited better wash fastness values than those of fabrics dyed by low energy disperse dyes. Because high energy class disperse dyes have higher molecular weight and show better wash fastness.

In this study, one bath dyeing of PBT/Nylon 6.6 fabrics was investigated. Therefore, only disperse dyes and their combinations such as disperse dyes – acidic dyes, disperse dyes – metal complex dyes were compared to obtain the best one bath dyeing process. Moreover, some dye combinations were recommended. It is seen that, one bath dyeing can be made for PBT/Nylon seamless products. In this section a comparison is made of how much water and energy would be consumed in two dyeing baths instead of these one bath dyeings. The calculations were made for 25 kg capacity machine. Comparison of water and time consumptions of the one and two bath dyeings are given on the Figure 8.

Figure 8: comparison of total water consumption and dyeing durations of one and two bath dyeings

As seen on Figure 8, two baths dyeing consumes more water and time than one bath dyeing. And also one bath dyeing's fastness values are moderate to good and commercially acceptable. It is noticed that, considering the difference between two dyeing one bath dyeing appears to be advantageous. In recent days when water consumption is becoming more important, the one bath dyeing technique becomes even more important. Additionally, the cost of the 1 kg of fabric dyed both one and two baths is given in the Table 9.

Table 9: the costs and cost reduction of the 1 kg of fabric dyed in one and two baths

Costs	Two baths dyeing	One bath dyeing	Reduction %
Labour costs	0.50 €	0.45 €	10
Maintance costs	0.09 €	0.08 €	11
Mechanical +illumination costs	0.19 €	0.16 €	15
Dye+Chemical costs	0.60 €	0.48 €	20

One bath dyeing is more advantegous than two baths dyeing. As shown on table 9, there is a decrease in the costs per 1 kg fabric. When all the results are examined, it is much more advantaneous to dye PBT/Nylon blended seamless fabrics in one bath instead of two baths.

V. CONCLUSIONS

Poly (butylene terephthalat) (PBT) fibers are important aromatic polyesters known as their good elasticity and easy dyeing properties. Nylon 6,6, fibers are the another common synthetic fibers, has extensive use in many areas thanks to its excellent physical properties. Generally PBT and nylon fibers are used in intimate, ready-to-wear, active and sportswear apparels, swimwear. Seamless technology is capable to meet the needs of consumers with performance, functionality and comfort and improves the aesthetic value and comfort, fredom of body movement, gives softness to the garment and minimizes the surface friction of the seams. PBT and nylon fibers can be used in seamless sport wears, tights and intimates. In this study, PBT/Nylon blended knitted seamless fabrics were dyed with disperse, acid and metal complex dyes at 98ºC in one bath. The color strength, colorimetric properties and fastness properties (rub and wash fastness) of these fabrics were investigated. All samples dyed with disperse and metal complex dyes exhibited better colorimetric properties than that of fabrics dyed with only disperse dyes or metal complex dyes. These dyed fabrics showed highest color strength values. The highest color strength value is 10,3 and has been observed on dyeing PBT/PA fabrics by metal complex and low energy disperse dyes with red color shades. All dyed samples exhibited excellent wet and dry rub fastness properties. Wash fastness values are generally exhibited good to excellent wash fastness by 5 grey scale rating for staining except nylon 6.6 and acetate. In terms of costs, water consumptions and all the results, it is much more advantaneous to dye PBT/Nylon blended seamless fabrics in one bath instead of two baths.

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RESEARCH ARTICLE / ARAŞTIRMA MAKALESİ

Antibacterial Characteristics of Nanofiber Structures Obtained by Benzalkonium Chloride Additive Poly (Vinyl Alcohol)/Gelatin

Benzalkonyum Klorür Katkili Pva/Gelatin Nanolif Yapilarinin Antibakteriyel Özellikleri

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Abstract

Polyvinyl alcohol (PVA) is a hydrophilous and semi-crystallized. It has attracted much relevancy due to its pretty chemical endurance, fine thermal determination, decent physical specialities, wonderful bio-compatibility and cheapness. Gelatin (G) is a natural polymer and that are interesting materials for biomedical applications. Electrospinning is a simple method that provides very porous nanofiber production with high surface area. It is possible to produce biomedical, filtration, energy storage and protective materials by using electrospinning method. Benzalkonium chloride (BAC) is a kind of anti-microbial cationic surface-active agent, which has been pretty used in merchant wound dressings and has a powerful status toward Gr+ bacteria.

In this study, nanofibers were produced from electrospinning of BAC, PVA and G containing solutions at various concentrations. The fibers of obtained nanofiber structure were uniform, continuous and intensive. The optimum parameters in terms of good mechanical and antibacterial properties were determined. S2 sample, electrospun from 11.63% PVA + 1.0% G +1.0% BAC containing solution, had the best morphological and mechanical properties due to having the thinnest fiber diameter (51±13nm) and the highest vertical strength (4.299MPa) and horizontal strength (4.058MPa). It also had antibacterial activity against all the bacteria tested (*E. coli, P. aeruginosa, B. subtilis and S. aureus*). Due to owning good mechanical and antibacterial properties, S2 sample can have many uses in medical sector.

Keywords: Electrospinning, poly(vinyl alcohol), gelatin, benzalkonium chloride, antibacterial, nanofiber

Öz

Poli (vinil alkol) (PVA), iyi kimyasal dayanımı, iyi termal kararlılığı, iyi fiziksel özellikleri, mükemmel biyouyumluluğu ve ucuzluğu nedeniyle çok dikkat çeken hidrofilik, yarı kristalli bir polimerdir. Jelatin (G) biyomedikal uygulamalar için ilginç ve doğal bir polimerdir. Elektroeğirme, yüksek yüzey alanına sahip çok gözenekli nano elyaf üretimi sağlayan basit bir yöntemdir. Elektrospinning yöntemi kullanılarak biyomedikal, filtrasyon, enerji depolama ve koruyucu malzemeler üretmek mümkündür. Benzalkonyum klorür (BAC), ticari yara sargısında yaygın olarak kullanılan ve Gram-pozitif bakterilere karşı güçlü bir role sahip olan bir tür antimikrobiyal katyonik yüzey aktif malzemedir. Bu çalışmada, çeşitli konsantrasyonlarda BAC, PVA ve G içeren çözeltilerin nano lifler üretilmiştir. Elde edilen nano lif yapısının lifleri homojen, sürekli ve yoğundur. İyi mekanik ve antibakteriyel özellikler açısından optimum parametreler belirlenmiştir. 11.63% PVA + 1.0% G +1.0% BAC içeren S2 örneği en iyi morfolojik ve mekanik özellikler göstermesinin yanında en ince elyaf çapına (51 ± 13nm) ve en yüksek dikey dayanıma (4.299MPa) ve yatay dayanıma (4.058MPa) sahiptir. Ayrıca test edilen tüm bakterilere (E. coli, P. aeruginosa, B. subtilis ve S. aureus) karşı antibakteriyel aktiviteye sahiptir. İyi mekanik ve antibakteriyel özelliklere sahip olması nedeniyle, S2 örneğinin tıbbi sektörde birçok kullanımı olabileceği sonucuna ulaşılmıştır.

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Anahtar Kelimeler: Elektroeğirme, PolivinilAlkol, PVA, Benzalkonyum Klorür, BAC, Antibakteriyel, Nanolif

I. INTRODUCTION

Electrospinning is a basic technique that dissolved materials is processed into nano-scale and micro-scale continuous fibers [1]. A typical electrospinning comprise of mainly three components: a capillary tube with pipette or needle of small diameter, a high voltage supplier and a metal collecting screen. There are four different regions within electrospinning process: the base region, the jet region, the splay region, the collector region [2]. The various synthetic polymers, natural polymers and a blend of both including proteins are used in the electrospinning process [3].

Polyvinyl alcohol (PVA) is a hydrophilous and semi-crystallized. It has attracted much relevancy due to its pretty chemical endurance, fine thermal determination, good physical specialities, wonderful bio-compatibility and cheapness. When diameter sizes of polymeric fibers reduce from micro-meter up to nano-meter variety. It has appeared several amazing properties. For example high surface area per bulk or per gob ratio, size probability for area operationalization and enhanced mechanical productivity due to a development in the constructional organization. These amazing specialities make superfine electrospinning polymeric fibers great applicants for many significant implementation, such as filtration, consolidating materials, wound dress, tissue scaffold, releasing of drug, etc [4].

Gelatin (G) is a connatural polymer. It can be popularly found in muscle, skin and bone of animal. Therefore, it has bio-degradability and bio-compatibility qualities. Gelatin has been jointly used in biomedical implementations. For example, wound healing and tissue scaffolds [5]. These apparent advantages of gelatin make it a goal constituent to enhance protein based constructs with similar mechanical strength to extracellular matrix (ECM) [6]. Also, gelatin is a promising choice for generation nano fiber due to it is inexpensive and present [5].

Benzalkonium chloride is a kind of anti-microbial cationic surface-active agent belonging to quaternacy ammonium compounds (QACs) with long alkyl chains of C_8 to C_{18}. It has antimicrobial activity against many microorganisms especially Gram-positive bacteria [7]. Due to its high antimicrobial activity, it is used extensively as biocides at hospitals and food procedure industries, and personal care products. Also, it is used at wound dressings in medical textile industries. Moreover, it is also environmentally friendly. During the waste-water treatment procedures, most BACs are generally removed by bio-degradation in assembly with adsorption on sewerage trash and the rest is drained in the effluence [8].

There are lots of studies about antibacterial activity of BAC containing membranes produced via electrospinning. For example; You et al. (2006) attached BAC to a solution of poly Lactic-co-Glycolic Acid (PLGA) for electrospun and obtained several diameters of BAC-PLGA nano fibers [9]. Wang et al. (2011) dissolved BAC in a polymer solution of poly Hydroxybutyrate – co-Hydroxyvalerate to enhance the conductivity at the electrospinning procedure [10]. Kim et al. (2007) prepared anti-microbial polycarbonate (PC) nano fibers using BAC as an anti-microbial spy and they stated that the BAC-PC nano fibers had fine anti-microbial activity toward Gr+ (S. aureus) and Gr – (E. coli and K. pneumonia) bacteria [11]. Electrospinning BAC – PVA nano fibers have been beforehand equiped by Arumugam et al. (2009), who put to use BAC as a conductive ingredient to a solution of PVA in electrospinning [12].

Addition of gelatin in PVA/BAC containing elecrospun membrane may add versatile properties of gelatin especially in terms of biocompability. There is no studies on production and anti-microbial activity of electrospinning BAC-PVA-G nanofibers. This study aims production of electrospun BAC-PVA-G nanofibers and optimization of the process in terms of mechanical and antibacterial properties.

II. MATERIALS AND METHODS

2.1 Materials

Polyvinyl alcohol (PVA) with molecular weight of 70,000 $gmol^{-1}$ and degree of hydrolysis of 85%, was supplied from Merck. Gelatin powder (bovine gelatin, 250 Bloom) was supplied from Alfasol. Alkyldimethylbenzylammonium chloride (benzalkonium chloride) (50%) was purchased from Kimetsan. The chemicals were used as received.

Main cultures of Gr – bacteria (*Es. coli* ATCC 35218 and *Ps. aeruginosa* ATCC 27853) and Gr+ bacteria (*Ba. subtilis* ATCC 6633 and *St. aureus* ATCC 25293) were supplied from Microbiologics.

2.2. Methods

2.2.1. Preparation of PVA/G/BAC electrospinning solution

Aqueous 12% (w/w) PVA solution was prepared by gently stirring for 2 hours at 70ºC. After formation of homogenous solution, gelatin powder was added, and the resulting mixture was stirred at 70ºC for 2 hours to make homogenous solution. Finally, various amount of BAC was added to produce the spinning solutions. The final concentrations of the electrospinning solutions were listed at Table 1.

Table 1: The final concentrations of the sample solutions for electrospinning. The concentrations were adjusted to 100% with distilled water.

Sample code	PVA (%)	Benzalkonium chloride (%)	Gelatin (%)
S1	11.75	0.5	1.0
S2	11.63	1.0	1.0
S3	11.27	2.5	1.0
S4	11.03	3.5	1.0
S5	10.79	4.5	1.0

2.2.3. Electrospinning

A 10 ml plastic syringe was filled with PVA/G/BAC solution for electrospinning. Inovenso NE300 Nanospinner model electrospinning device was used to produce nanofibers. A high voltage power was applied to generate the high electric field between the nozzle and the cylindrical collector. Cylindrical collector surface was covered with grease-proof paper. The set parameters of electrospinning process were listed at Table 2.

Table 2: Electrospinning parameter

Parameter	S1	S2	S3	S4	S5
Applied Voltage (kV)*	36	36	39	30	27
Feeding Speed (ml/h)	1	1	1	0.7	0.7
Velocity of cylindrical rotating collector (rpm)	100	100	100	100	100

* The applied voltage was adjusted according to the solution conductivity.

2.2.4. Viscosity and conductivity of solutions

Viscosities of the polymer solutions were determined by using viscometer (Brookfield DV-E Viscometer, USA) with S21 spindle at 50rpm and 60rpm. Conductivities of the polymer solutions were gauged by conductivity meter (WTW Cond 3110, Germany). All experiments were fulfiled out at room temperature.

2.2.5. Morphology of nanofiber structure

The morphologies of the electrospinning nanofibers were analyzed with SEM images (JEOL JSM-5910 LV, Japan). The average fiber diameters of the PVA/G/BAC nanofibers were measured by Image J software from the SEM images.

2.2.6. Mechanical test

All nanofiber membranes were cut to 50x10mm (length x width) for the mechanical test. Instron 4411 universal test device was used to examine the mechanical properties of nanofiber membranes. The piston speed was set 30mm/min.

The thicknesses of nanofiber membranes were measured with a Mitutoyo digital thickness gauge.

2.2.7. Antibacterial activity

ISO 20645:2004 method was simulated to specify the anti-bacterial activity of electrospun mats [13]. The bacterial strains preserved at 80°C were pre-cultured in 10ml liquid medium in waging incubator at 37±1°C for 16h. The liquid media were Nutrient Broth for *E. coli* and *B. subtilis* bacteria, and Trypticase Soy Broth for *S. aureus* and *P. aeruginosa* bacteria. 10µl of the pre-cultures were vaccinated into the requested capacity of fresh liquid media. Proximate CFU numbers were guessed from McFarland densitometer measurement and the bacterial culture was generated by incubating at 37±1°C till the bacteria concentration arrived 10^8 CFU/ml [14].

To test anti-bacterial activities of the mats, soft agar plaques were made by adding 7.5g/L Agar into the broth media described above. After cooling to 40-45 °C, the bacterial culture was added such a way that bacterial concentration reaches to 10^6 CFU/ml, which was adjusted via McFarland measurement. After gelation of the agar mediums, each mat to be tested was cut in 20mm x 20mm size and was placed on the soft agar medium. The petri plates were incubated at 37±1°C for 24 hours. For accuracy, the tests were executed and rehearsed three times. Then, the inhibition zones of S1-S5 samples were compared with each other [15].

III. RESULTS

3.1. Viscosity And Conductivity Of Solutions

Table 3 shows viscosity and conductivity of all solutions.

Table 3: Viscosity and conductivity of the solutions

Number	Viscosity values of solutions (cP)	Conductivity values of solutions (mS/cm)
S1	322	1645
S2	348	1935
S3	414*	3000
S4	344	3690
S5	616	4570

*Measured at 60rpm, while the other samples were measured at 50rpm.

As shown at Table 3, solution viscosities generally increased in parallel to increase in BAC concentration and the highest viscosity was observed at the most BAC including sample. Increase in BAC concentration increased the conductivities of the solution and the highest conductivity was observed at the most BAC including sample. Increase in the conductivity made it necessary to reduce the applied voltage at S4 and S5.

3.2. Morphology of Nanofiber Membrane

The morphologies of the electrospinning nanofibers were analyzed with SEM images.

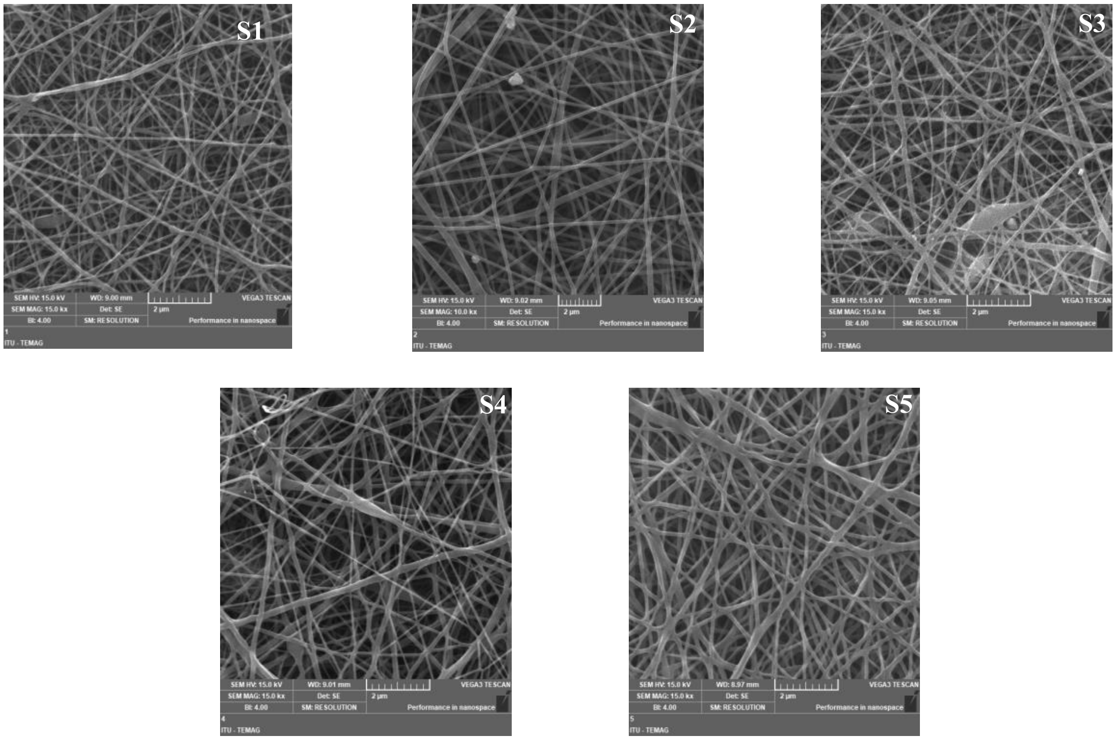

Figure 1: The SEM images of S1-S5 samples.

The fiber diameters of samples S1-S5 were 51±13nm, 135±19nm, 67±18nm, 82±18nm and 93±13nm respectively. When BAC concentration increased, the nanofiber diameters of PVA/G/BAC membrane increased. But, the average nanofiber diameter increased at 1.0% BAC concentration (S2). The thinnest fiber diameter (51±13nm) was observed at the least BAC containing sample (S1).

3.3. Mechanical Test

Vertical and horizontal strength values of PVA/G/BAC nanofiber membranes were measured (Chart 1 and Chart 2).

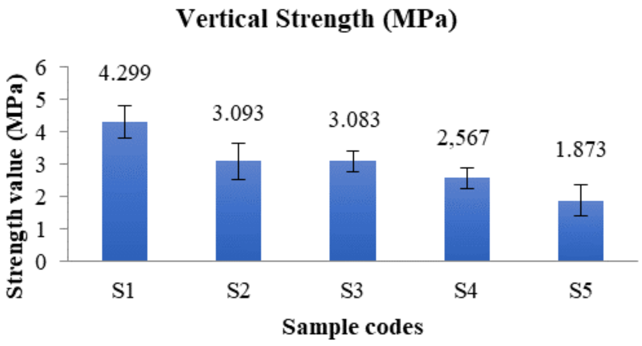

Chart 1: The measured vertical strength values of nanofiber membranes

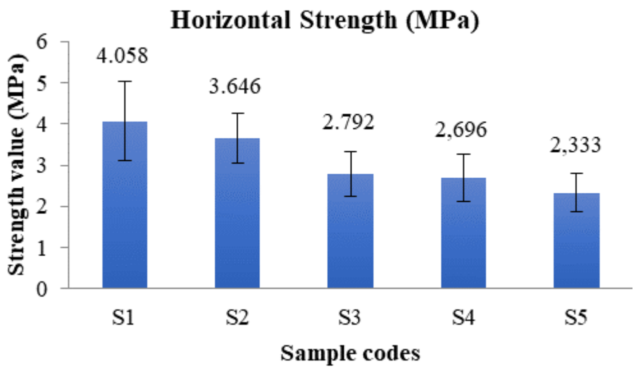

Chart 2: The measured horizontal strength values of nanofiber membranes

The highest vertical strength and horizontal strengths were observed at the least two BAC concentrations (0.5% at S1 and 1.0% at S2). When BAC concentration increased, vertical and horizontal strengths decreased gradually.

The thicknesses of nanofiber membranes were also measured (Chart 3).

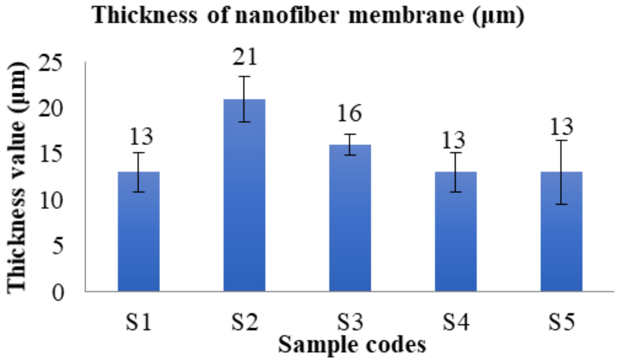

Chart 3: The thickness values of nanofiber membranes

The thickest membrane (21mm) was observed when BAC concentration was 1% (S2). The thicknesses (13mm) of the membranes were close the each other at S1, S4 and S5 samples.

3.4. Antibacterial Activity

Due to the water solubility of PVA, the membranes dissolved in the agar medium and effective samples created inhibition zones. The most resistant bacterium was *P. aeruginosa,* which was not inhibited by S1, produced from 0.5% BAC containing solution (Figure 2).

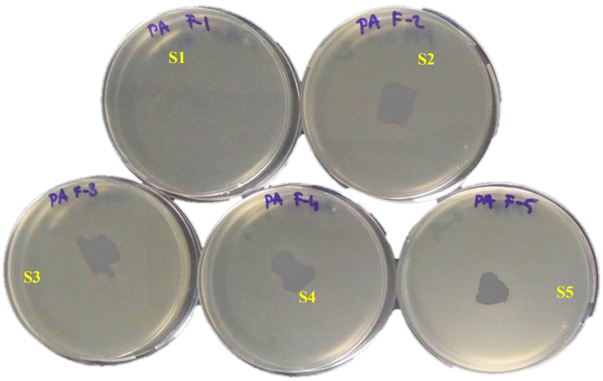

Figure 2: Antibacterial activity of S1-S5 samples against *P. aeruginosa*.

As shown at Figure 2, S1 sample did not create an inhibition zone against *P. aeruginosa*. The minimum BAC concentration that produced an inhibition zone against *P. aeruginosa* was 1% (S2). S2 sample produced inhibition zone against all the bacteria tested (Figure 3). Therefore, among the tested samples (S1-S5), S2 sample with minimum amount of chemicals seems optimum for inhibition of all the bacteria tested.

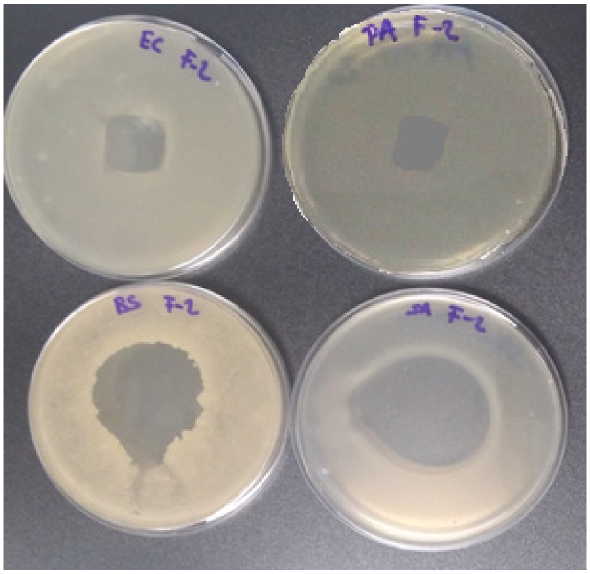

Figure 3: Antibacterial activity of S2 against *E. coli* (EC), *P. aeruginosa* (PA), *B. subtilis* (BS) and *S. aureus* (SA) bacteria.

IV. CONCLUSION

In this study, PVA/G/BAC aqueous solutions were electrospun to produce nanofiber structure with high mechanical strength and with antibacterial activity against Gr – (*E. coli* and *P. aeruginosa*) and Gr+ (*B. subtilis* and *S. aureus*) bacteria. Viscosities of the solutions generally increased as increase in BAC concentration. Also, conductivities of the solutions increased in parallel to increase in BAC concentration.

The membranes produced from electrospinning of PVA/G/BAC aqueous solutions had uniform and intense nanofiber distribution with fiber diameter of 51±13nm-135±19nm. The highest vertical and horizontal strengths nanofibers belonged to S1 and the order of the strengths were S1>S2>S3>S4>S5. Antibacterial activities of the samples showed that all the samples were effective against *E. coli, B. subtilis* and *S. aureus* but S1 sample (including 0.5% BAC) was ineffective against *P. aeruginosa*. S2-S5 samples were effective toward all the bacteria tested. 1.0% BAC (S2) was the minimum concentration for inhibition of all the bacteria tested. Therefore, due to its second best mechanical properties and its low BAC concentration (1.0%), which decreases environmental concerns and production costs, S2 sample (11.63% PVA + 1.0% G +1.0% BAC) was thought ideal for electrospinning of PVA/G/BAC solution system.

The membranes electrospun from S2 solution can be good candidates for medical applications like roll bandage for its good mechanical properties and antibacterial activities against common bacteria (*E. coli* and *B. subtilis*) and pathogenic bacteria (*S. aureus* and *P. aeruginosa*) causing nosocomial infections and wound infections.

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