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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.

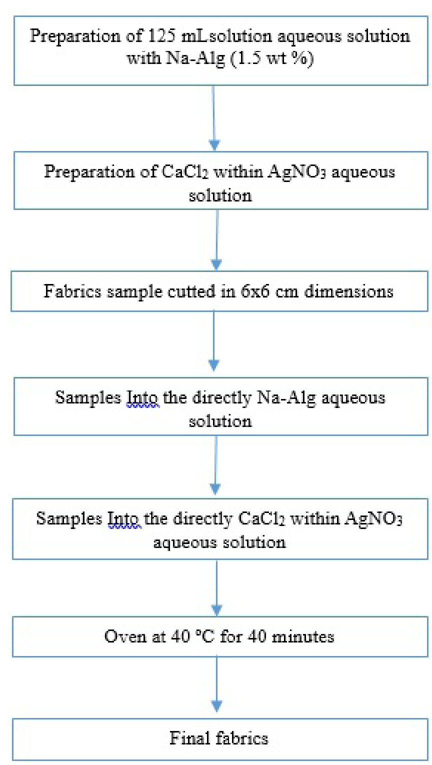
In a typical synthesis 125 mL of Na-Alg (1.5 wt %) was added to a 125 mL aqueous solution. Following that, exactly 1.87 gr. Na-Alg was mixed in the totally 125 ml aqueous solution at 70 °C for 100 minutes in a 1600 rpm. Together with this process, on the other side $AgNO_3$ (0.05 M) solution were prepared.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

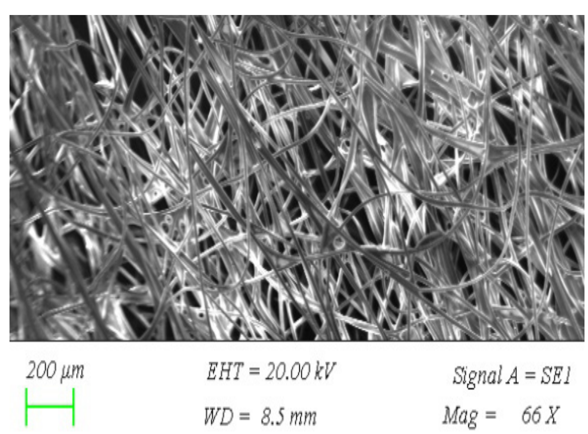
3.2 Antibacterial Analysis

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.

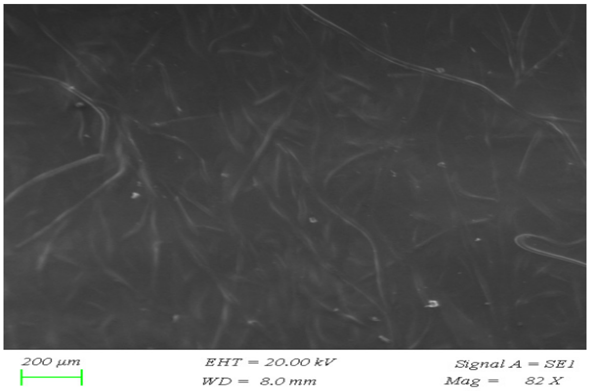
IV. RESULTS AND DISCUSSIONS

4.1 Results of SEM

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



a



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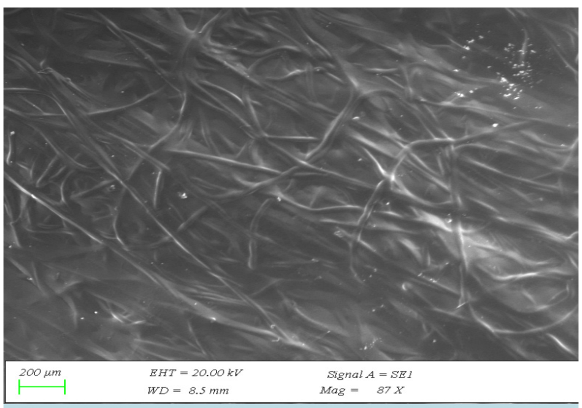

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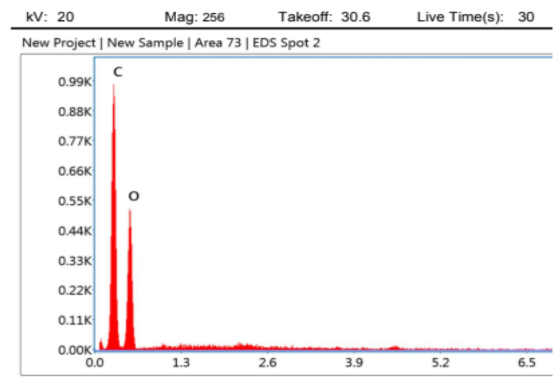
Figure 1. SEM results of (a) sample 1, (b) sample 4 and (c) sample 7

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

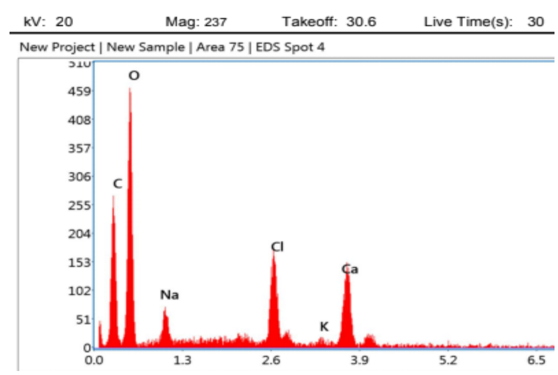
Edx analysis gave the content of elementes in the chemical structure of materials. Totally, nine different fabric samples were analyzed in EDX instrument (ZEISS/EVO). When references samples were investigated, it was seen there were C and O elements were presented and while ratio of viscose in the fabric 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

a



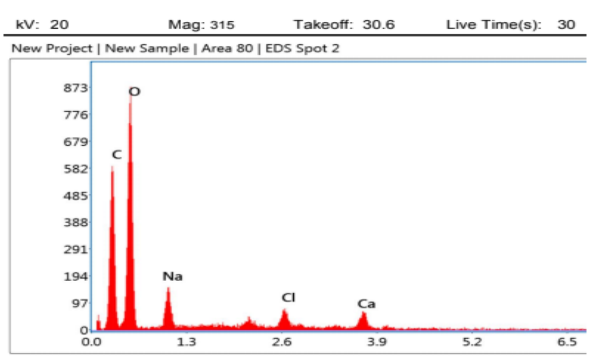
Smart Quant Results

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

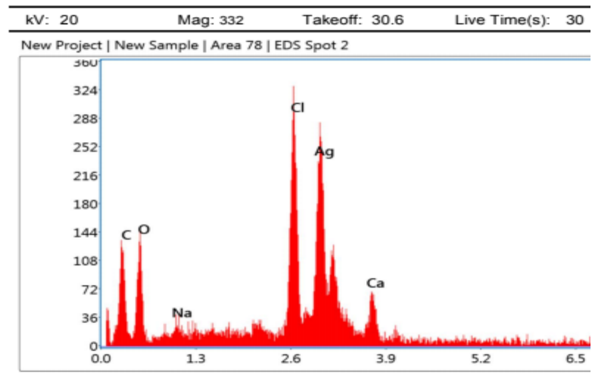
Finally, EDX results of samples appliquéd with $AgNO_3$ were investigated. According to edx results, there were not seen any Ag ions onto the samples with 40% viscose to 60% polyester content. However, as opposed to this result, onto the samples with 70% viscose to 30% polyester content and samples with 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

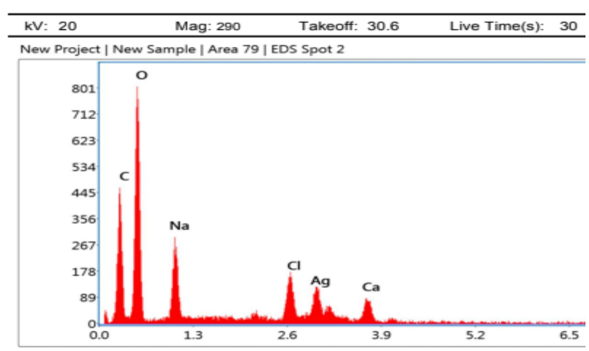
a



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

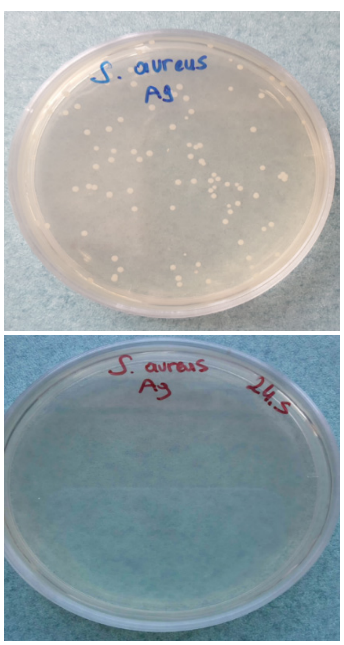


Figure 4. Bacterial growth in a (zero contact time) and b (24 contact time) for Sample 7 on *S. aureus*

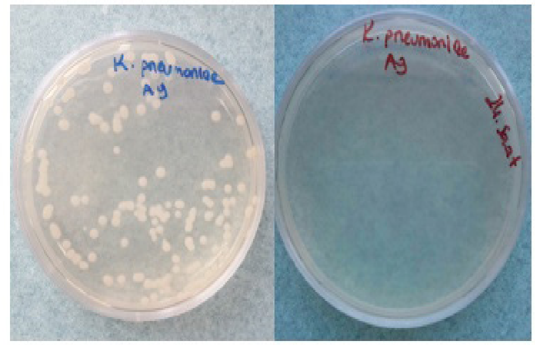


Figure 5. Bacterial growth in a (zero contact time) and b (24 hours contact time) for Sample 7 on *K. pneumoniae*

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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