Study of Dyeing and Obtaining Antimicrobial Effects of Virgin and Recycled Polyester Fibers

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In this paper we studied the dyeing behaviour of polyester fibers from recycled polyester and virgin dyed with Foron brilliant rot SGL (CI Disperse Red 121). The amount of dye fixed recycled fiber is higher than that of virgin. Elemental analysis confirms a slight change in the carbon and oxygen content of recycled fiber compared to virgin. Thus the recycled polyester fiber carbon content is higher than virgin fiber in oxygen exchange is less. Dyed samples were treated with $AgNO_3$ in order to confer antibacterial properties. The analyses SEM/EDAX have highlighted the presence of silver ions on the surface of fibers.

Keywords: dyeing, recycled PET, virgin PET, disperse dye, antimicrobian effect

The polyethylene terephthalate obtained from recycling packages represents a potentially inexpensive source of raw matter. The main advantage of recycled PET consists in the fact that it does not require reparation from other materials and dyeing agents. The fibers obtained from recycled PET have some of the most various uses (fig. 1) [1-7].

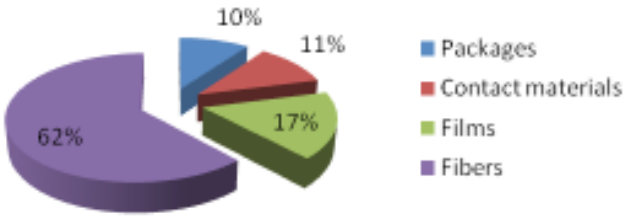

Fig. 1. Intended uses of recycled PET

The fibers and strands derived from recycled PET (either individual or combined with natural fibers) are used as raw materials for carpets, upholsteries, toys, as insulation for clothing or sleeping bags, apparel and geotextiles, films, adhesive bandages and obtain the "glass within glass" process [8-13]. Currently, the largest quantity of polyethylene terephthalate is used in the textile industry.

Soon, we will be able to yield geotextile materials entirely from polyethylene terephthalate provided that an adequate level of quality and a constant, industrial scale production are ensured.

Besides the usual applications PET can be used to obtain multifunctional materials. The PET may be functional with different antimicrobial treatments which are based on metal ions Ag^+, Cu^{2+}, Ti^{2+}.

Since ancient times, the silver ion has been known to be effective against a broad range of microorganisms. Silver destroys cell membranes, distorting protein, inhibits enzyme activity and lipid synthesis which are essential for the survival of microorganisms [14-18].

In this paper is studied the tinctorial activity of virgin and recycled PET materials. For optimal dyed samples (3% Disperse Red 121 dye for 30 min) was performed an antimicrobial treatment with 18 mg $AgNO_3$/g fiber.

Experimental part

Materials

The recycled PET fibers were provided by GreenFiber International, Buzău. The recycled and virgin PET fibers (17 dtex fiber size) used for experimentations were degreased under the following conditions: 5g/L Lavotan DSU, T - 60°C, t - 60 min.

The main characteristics of the fibers used in dyeing polyester are shown in table 1.

The process of dyeing the polyethylene terephthalate samples

The virgin and recycled polyethylene terephthalate samples were dyed with the C.I. Disperse Red 121 (fig. 2) under the following conditions: 0.2; 0.6; 1; 1.4; 2 and 3% dye, respectively, in reference to the amount of material, 1% CH_3COOH, Hm 50:1, at 130°C for 5, 10, 20, 30 and 60 min, respectively, using the Polycolor P 4702 type machine. After dyeing, the samples were thoroughly rinsed with hot and cold water.

Dyeing evaluation

Determining the dye amount bound to the fiber

In order to determine the amount of dye bound to fiber was established the standard curve for dye Disperse Red 121 dye. Equation given by standard curve is y = 22.992x,

Fiber	C (%)	O (%)	Crystallinity (%)
Recycled PET	49.87	50.13	34.72
Virgin PET	54.16	45.85	36.32

Table 1
PET FIBER CARACTERISTICS

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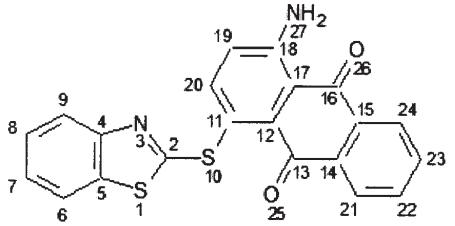


1-amino-4-(1,3-benzothiazol-2-ylsulfanyl)anthracene-9,10-dione

Fig. 2. Disperse dye structure

where x is the concentration of the dye solution, and y is the absorbance at wavelength at which absorption is maximized. The absorbances were measured on a UV-Vis spectrophotometer. Using the standard curve were calculated amount of dye from the dyeing bath waste (Wr), and the amount of dye in the resulting wash water (V). The amount of dye bound to the fiber (Wf) was calculated as the difference between the initial quantity of dye (Wi) and the quantities of dye Wr and Ws:

$$W_f = Wi_. - (W_r + W_s) \quad (1)$$

Determination of colour strenght (K/S)

The colour strenght of the samples dyed with the disperse dye was assessed using the K/S index determined with the Spectroflash 300® spectrophotometer produced by DATACOLOR for the D65/10 illuminant [19-29]. The correlation between the dye concentration and the remission of the dyed samples is provided by the relation:

$$A \cdot c = f(R) \quad (2)$$

where: A – a constant depending upon the dye, the textile carrier, the wave length, as well as the conditions under which the dyeing was performed; c – represents the dye concentration in the dyed material; f(R) – is a dyeing remission function that may be established both by theoretical, as well as empirical means. For practical purposes, colour strenght measurement uses the f(R) function, theoretically inferred by Kubelka and Munk [19-22]:

$$K/S = (1-R)^2/2R \quad (3)$$

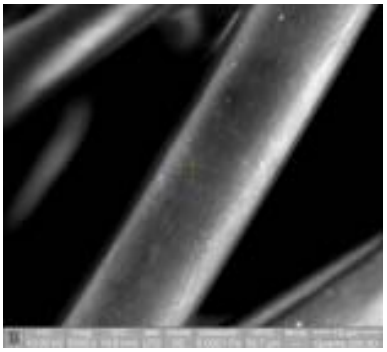
where: K – represents a light absorption coefficient; S – light diffusion coefficient.

$AgNO_3$ treatment

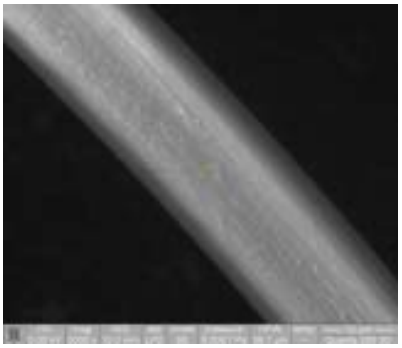
In order to confer antimicrobial properties of dyed polyester materials, they were treated with 18 mg $AgNO_3$/ g fiber at 100^{0}C for 30 min. After the treatment, the samples were rinsed thoroughly with distilled water.

SEM/EDX analysis

A QUANTA 200 3DDUAL BEAM electron microscope was used, which is a combination of two systems (SEM and FIB), by whose means, by sending an electron beam on the treated samples, three-dimensional images could be obtained, with a magnification of 100,000X. Moreover, by using the X radiation with dispersive energy (EDX), the elemental analyses were possible for the identification of the surface characteristics and a high resolution chemical analysis.



Virgin PET



Recicled PET

Fig. 3. SEM analisys

Antimicrobial test

Antimicrobial test was carried out on dyed virgin and recycled PET fabrics. The dyed samples retreated with $AgNO_3$ were tested against gram positive bacteria (*Staphylococcus aureus, Bacillus subtilis*) and gram negative bacteria (*Pseudomonas aeruginosa, Escherichia coli*). The sensibility of bacteria against treated samples was tested „in vitro" under optimal and standardized conditions of inoculation. For this purpose the Kirby-Bauer disc diffusion method was used [30, 31].

Results and discussions

Morphological characterization of the virgin and recycled PET fibers are emphasized by SEM photos.

The analysis of SEM photos does not highlight essential changes of the surface.

Evaluation of dyeing

The efficiency of dyeing was assessed both quantitatively, by determining the amount of dye bound to the fiber, and qualitatively by colour intensity measuring.

Determining the dye amount bound to the fiber

The amount of dye present on the polyethylene terephthalate fibers was calculated according to relation 1 and is presented in tables 2 and 3.

Dye concentration mg dye)	Amount of dye bound to the fiber (mg dye/g fiber)				
	5 min	10 min	20 min	30 min	60 min
2	1.28	1.53	1.61	1.76	1.78
6	2.08	3.07	3.71	5.36	5.42
10	2.76	4.29	6.91	8.00	8.29
14	3.91	7.09	9.28	10.92	11.82
20	6.02	9.86	14.01	16.25	17.34
30	9.17	12.94	19.29	25.47	26.39

Table 2
THE AMOUNT OF DISPERSE RED 121 DYE BOUND ON VIRGIN PET FIBER

Dye concentration (mg)	Amount of dye bound to the fiber (mg dye/g fiber)				
	5 min	10 min	20 min	30 min	60 min
2	1.41	1.69	1.78	1.94	1.96
6	2.29	3.38	4.08	5.9	6.6
10	3.04	4.72	7.6	8.8	9.12
14	4.3	7.8	10.2	12	12.99
20	6.62	10.84	15.4	17.86	19.06
30	10.08	14.23	21.8	27.95	29.01

Table 3
THE AMOUNT OF DISPERSE RED 121 DYE BOUND ON RECYCLED PET FIBER

Fyber tipe	Microorganism test			
	Staphylococcus aureus	Bacillus subtilis	Escherichia coli	Pseudomonas aeruginosa
Virgin PET	++	++	+	++
Recycled PET	+++	++	+	++
Untreated samples	-	-	-	-

Table 4
ANTIBACTERIAL EFFECT

+ weak inhibition of culture development; ++ apparent inhibition of culture development;+++ good inhibition of culture development; - lack of increase and development of bacteria

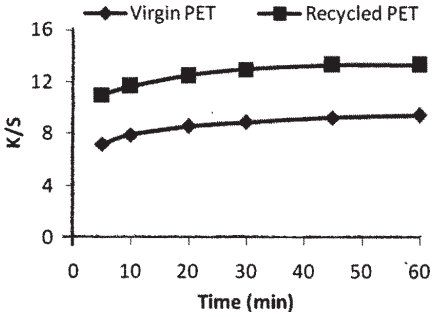


Fig. 4. Colour intensity variation depending on the dying time

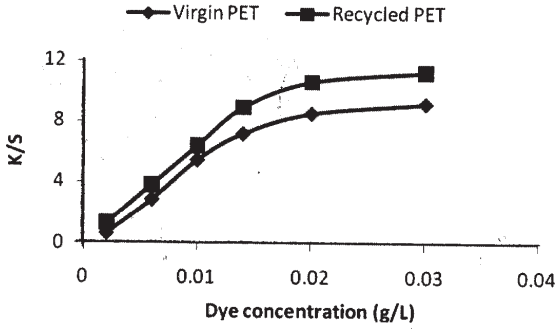


Fig. 5. Colour intensity variation depending on the dye concentration

According to the results presented in tables 2 and 3, the amount of dye bound to the polyethylene fibers increases with increasing the amount of dye in the dye bath. Comparing those two fibers results that the amount of dye bound to the recycled polyethylene terephthalate fiber is higher than the amount of dye bound to the virgin fiber.

Determination of color strenght (K/S)

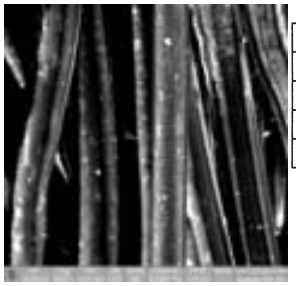
Comparing those two fibers, one can see that the variation of colour intensity for the two types of fibers dyed with Disperse Red 121 dye at different periods of time and different concentrations is shown in figures 4-5.

Analyzing the results obtained, we found that colour strenght (K/S) increases at the same time with the increase of the dye concentration in the treatment bath. The result produced by the comparative analysis of the tinctorial capacity displayed by the fibers derived from recycled PET and those derived from virgin PET indicates that recycled polyethylene terephthalate has a higher dyeing capacity. Regarding the influence of the dying time it can be concluded that the amount of dye bound to fiber or colour intensity does not change significantly at the dyeing duration more than 30 min.

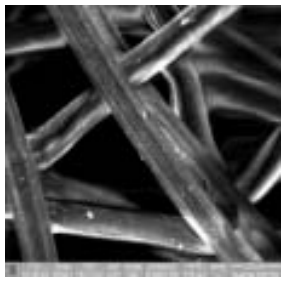
SEM/EDAX analysis for samples treated with $AgNO_3$

The samples dying with 3% dye for 30 min was treated with $AgNO_3$ solution. The presence of silver on the surface of the fibers was highlighted by SEM/EDX (fig.6).

The results concerning the antimicrobial activity are shown in table 4



Element	Wt%	At%
CK	48.86	57.73
OK	46.79	41.70
AgL	04.35	00.57
Matrix	Correction	ZAF



Element	Wt%	At%
CK	47.84	56.04
OK	48.31	42.63
AgL	03.85	01.33
Matrix	Correction	ZAF

Recycled PET
Virgin PET

Fig. 6. SEM-EDAX analisys

Antibacterial test

The antibacterial test was carried out on the samples of virgin and recycled PET, dyed with 3% CI Disperse Red 121 for 30 min and then treated with a solution of 18 mg of $AgNO_3$/g fiber. The results regarding antibacterial activity are presented in table 4.

The best results regarding the antibacterial activity against the Staphylococcus aureus bacteria are provided from the recycled PET samples. The obtained results are confirmed also by photographic images presented in figure 7.

Untreated sample Recycled PET

Fig. 7. The inhibition areas of treated and untreated recycled PET

The obtained results showed that both the virgin and the recycled PET samples, treated with $AgNO_3$, have antibacterial activity against gram positive and gram negative bacteria, as confirmed by the literature. A stronger inhibition of the growth and the development of microorganisms was obtained in the case of the bacteria *Staphylococcus aureus, Bacillus subtilis, Pseudomonas aeruginosa*, in comparison to the species *Escherichia coli*.

Conclusions

According to the results obtained in this paper, the optimal duration of polyetylentereftalat dyeing is 30 min. Increasing dyeing time over 30 min did not change significantly the quantity of dye fixed on the PET fiber. Both chromatic analysis parameters (colour intensity) and determine the amount of dye fixed on the fiber, showed that recycled polyethylene fiber is dyed more intensely than virgin PET. The virgin and recycled PET samples treated with $AgNO_3$, show antibacterial activity against gram positive and gram negative bacteria. The best antibacterial activity was obtained against gram positive bacteria Staphylococcus aureus.

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