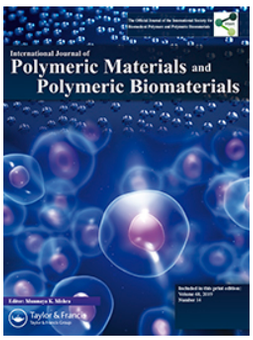

International Journal of Polymeric Materials and Polymeric Biomaterials

ISSN: 0091-4037 (Print) 1563-535X (Online) Journal homepage: https://www.tandfonline.com/loi/gpom20

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To cite this article: Metin Arslan & Kübra Günay (2019) Synthesis of modified poly(ethylene terephthalate) fibers with antibacterial properties and their characterization, International Journal of Polymeric Materials and Polymeric Biomaterials, 68:14, 811-818, DOI: 10.1080/00914037.2018.1506987

To link to this article: https://doi.org/10.1080/00914037.2018.1506987

Published online: 10 Oct 2018.

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INTERNATIONAL JOURNAL OF POLYMERIC MATERIALS AND POLYMERIC BIOMATERIALS
2019, VOL. 68, NO. 14, 811–818
https://doi.org/10.1080/00914037.2018.1506987

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Synthesis of modified poly(ethylene terephthalate) fibers with antibacterial properties and their characterization

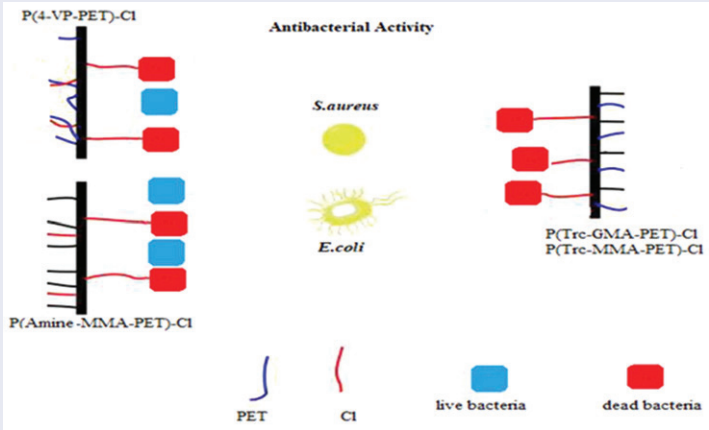
Metin Arslan[a] and Kübra Günay[b]

[a]Department of Chemistry and Chemical Processing Technologies, Kırıkkale Vocational High School, Kırıkkale University, Yahsihan, Kırıkkale, Turkey; [b]Graduate School of Natural and Applied Sciences, Kırıkkale University, Yahsihan, Kırıkkale, Turkey

ABSTRACT

Poly(ethylene terephthalate) (PET) fibers were grafted with vinyl monomers by utilizing benzoyl peroxide. Grafted PET fibers were modified in optimized conditions with several functional groups such as amine, chlorine, hydrogen peroxide, and triclosan to gain antibacterial feature. The second part of this study comprised examination of the antibacterial features of PET fibers via use of *Staphylococcus aureus* (ATCC 29213) and *Escherichia coli* (ATCC 25922) bacteria. Kirby-Bauer test is used to study antibacterial properties. The longest zone diameter for Trc-GMA-g-PET fibers was 56 mm for *E. Coli* whereas the biggest diameter for *S. aureus* bacteria was 130 mm with Trc-MMA-g-PET fibers.

GRAPHICAL ABSTRACT



ARTICLE HISTORY
Received 2 April 2018
Accepted 24 July 2018

KEYWORDS
Poly(ethylene terephthalate) fibers; graft polymerization; 4-vinylpyridine; methacrylic acid; glycidyl methacrylate; antibacterial properties

1. Introduction

Scientific studies are facilitated with the rapid development in the technology. Companies are competing to offer cheaper and long-lasting products to meet the expectations of the consumers. One of the fields experiencing this competition is the textile sector, which aims for more comfortable, more practical, cheaper, and, the most importantly, healthier fabric productions[1]. Industrial textile is the most dynamic sector of the textile and the ready-to-wear clothing industry, which has high growth rates in the recent years. Synthetic textile products that have a strategic role in the areas utilizing the advanced technologies such as aviation, military and health are used extensively in daily life including sports, ready-made clothing, home textile, furniture, building equipment, and automobile[2]. The textile products used in public places such as hospitals, kindergardens and hostels are carriers for the infectious pathogens[3, 4]. Thus, textiles contaminated with human pathogens and on which the microorganisms can propagate creates a great danger for the human health[5]. Therefore, the demand for the antibacterial textile products has increased. Antibacterial fibers are effective on microorganisms that both threaten the human health and decrease the fabric performance and comfort[6–8].

The most important field in which the synthetic fibers made up of synthetic polymers is the textile industry[9]. Polyesters, polyamides and polyacrylonitrile are the polymers that have gained high importance commercially in the fibers production[10–12]. Today, PET is almost the only polymer used in the polyester fibers production. The superior features of this product are having superior physical and chemical properties, being resistant to the bacteria, moths and light, and being resistant to the acidic compounds even at its boiling temperature[13–16].

CONTACT Metin Arslan marslan@kku.edu.tr Department of Chemistry and Chemical Processing Technologies, Kırıkkale Vocational High School, Kırıkkale University, Yahsihan, 71450 Kırıkkale, Turkey.
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The demand for the new antimicrobial textile products has increased in the recent years in the rapidly developing textile sector due to the danger of rapid spread of epidemic infectious diseases (e.g. flu)[17, 18]. Bacteria endanger health by multiplying fast on textile surfaces under optimum temperature, humidity, and other environmental factors. Nowadays, antimicrobial textile products are effective only against specific microorganism groups (such as bacteria) since they contain only one type of antimicrobial content[19, 20].

The aim of this study was to produce new materials having antibacterial properties. Accordingly, PET fibers were modified with 4-VP, MAA and GMA by graft copolymerization method. Then, HCl was bound to fibers over pyridine. After MAA was aminated, they were treated with chloride. Moreover, MAA and GMA were modified with triclosan, which is a strong chemical agent[21–24]. Compounds containing chlorine are the oldest disinfectants. They effectively kill most of the microorganisms including fungi and viruses. They also kill the bacteria and their spores resistant to acid at high concentrations with long-term induction[25].

Modified fibers that gained antimicrobial properties were characterized via Fourier-transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The antibacteial effect of the modified PET fibers on bacteria (*E. coli* and *S. aureus*) were evaluated. Antibacteial effect of the modified PET fibers were examined via Disc Diffusion Sensitivity Assay and liquid medium tests and eventually results were quantified in terms of bacterial growth curve.

2. Experimantal

2.1. Materials

All reagents and solvents were commercially available and used as received. PET fibers (122 dTex, middle drawing) used during graft copolymerization were obtained from SASA Co. (Adana, Turkey). The fibers samples were washed in Soxhlet Extractor with acetone due to a possible contamination during production or use[26] and fibers were used after they were brought up to a fixed weight. 4-VP was purified by vacuum distillation at 2 mmHg and 65 °C. 4-VP, MMA, and GMA were used as monomers during graft copolymerization.

Nutrient Broth Agar solid medium was used as Nutrient Broth liquid medium. As microorganism, Gram positive *S. aureus* (ATCC 29213) and Gram negative *E. coli* (ATCC 25922) were used.

2.2. Graft polymerization procedure

During the graf copolymerization of vinyl monomers on the PET fibers, 100 mL polymerization tubes with nitrogen gas inlet were used. PET fibers were placed in polymerization tubes including the monomer and water. Polymerization tubes were treated with nitrogen gas for 20 minutes in a water bath with constant temperature to reach thermal equilibrium. A total volume of 20 mL was obtained by the addition of 2 mL benzoyl peroxide (Bz_2O_2) solution in acetone at an appropriate concentration and graft polymerization was performed under a condenser at nitrogen atmosphere at certain periods of time. At the end of a certain graft polymerization period, fibers were taken out from the polymerization tubes and monomers and homopolymers on the fibers surfaces were washed away in Soxhlet Extractor by applying the appropriate solvents. Fibers were dried in the incubator and weighed[27].

Figure 1. Discs in agar medium (1) antibiotic disc, (2) empty disc, (3) antibacteial polymer.

The graft yield (G Y) was calculated gravimetrically from the differences between the weights of original and the grafted fibers and represented with the following formula:

$$G\ Y(\%) = [(g_A - g_0)/g_0] \times 100 \quad (1)$$

g_A = Dry weight of the grafted fibers
g_0 = Dry weight of the original fibers

2.3. Modification with oxidation and chlorination of 4-VP-g-PET fibers

For oxidation, grafted fibers were applied in 30 mL 20% hydrogen peroxide- acetic acid mixture in 50 mL conical flask. This solution was mixed in a shaker at 110 rpm for 24 h at 25 °C. Oxidized fibers were washed in water for 12 h and dried in incubator at 50 °C[28].

For chlorination, grafted fibers were applied in 30 mL 25% hydrochloric acid and water in 50 mL conical flask. This mixture was spinned at 110 rpm for 3 h at 25 °C in a water bath with shaker. Chlorinated fibers were washed with water for 8 h and dried in an incubator at 50 °C.

2.4. Modification of MMA-g-PET fibers

Amination, chlorination of aminated fibers, and binding of triclosan to the PET fibers grafted with MMA was performed.

For amination, hexamethylene diamine (HMDA) and tetraethylene pentamine (TEPA) were used as amines. Grafted fibers were applied in 30 mL 50% HMDA-ethanol and 50% TEPA-2-propanol mixture in 50 mL conical flask. This mixture was spinned at 110 rpm for 1 h at 30 °C in a water bath with a shaker. Aminated fibers were washed in methanol for 3 h and in water for 24 h and dried in incubator at 50 °C[29].

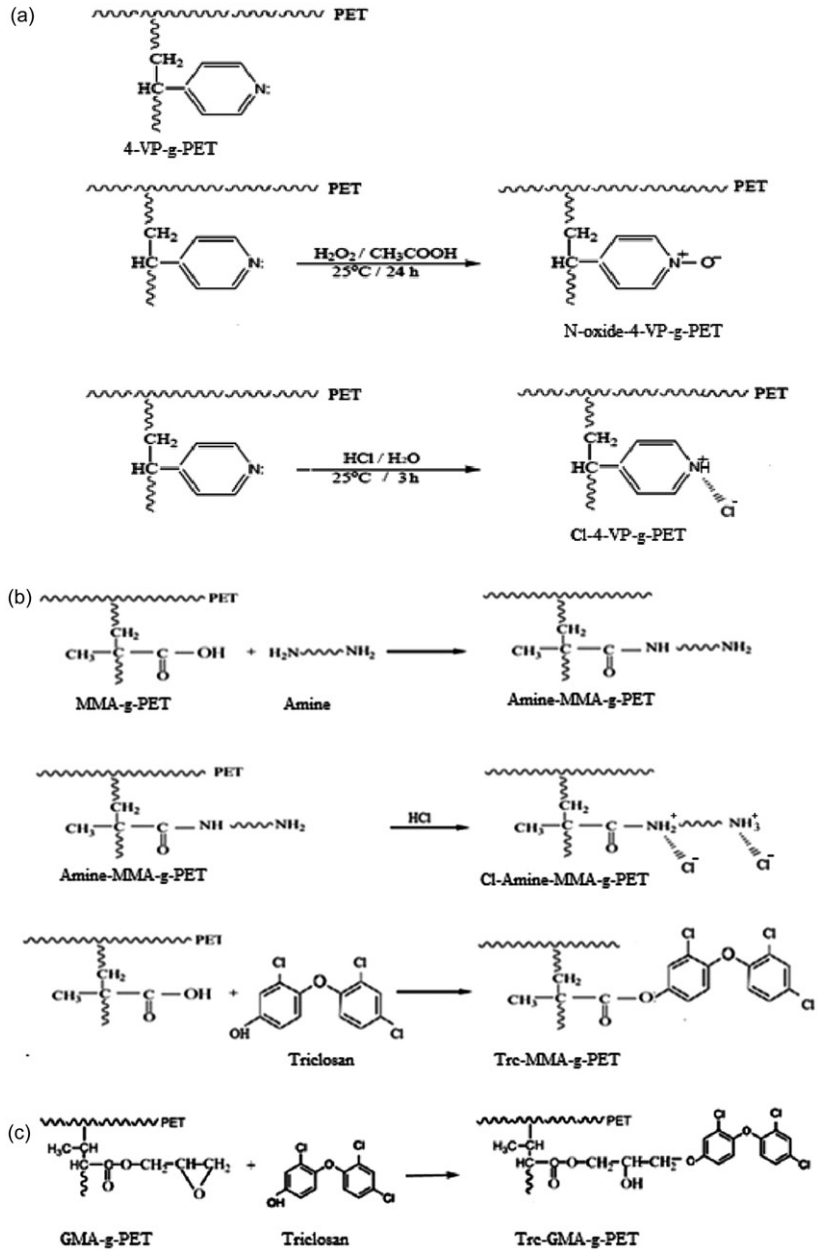

Figure 2. Modified PET fibers (a) synthesis mechanism of 4-VP-g-PET fibers and its derivatives (b) synthesis mechanism of MMA-g-PET fibers and its derivatives (c) synthesis mechanism of GMA and derivatives.

For chlorination, aminated fibers were applied in 30 mL 25% hydrochloric acid- water mixture in 50 mL conical flasks. This mixture was spinned at 110 rpm for 3 h at 25 °C in a water incubator (Selectra) with shaker. Chlorinated fibers were washed with water for 8 h and dried in an incubator at 50 °C.

For modification with triclosan, 9.15 mmol triclosan was dissolved in 100 mL tetrahydrofuran (THF). For acidic environment, 1 mL 0.1 M H_2SO_4 were added into 20 mL triclosan solution. The solution containing 0.1 g grafted fibers were spinned at 110 rpm for 12 h at 50 °C in a water bath with shaker. Fibers bound by triclosan were washed in water for 24 h and dried in an incubator at 50 °C.

2.5. Modification with triclosan of GMA-g-PET fibers

9.15 mmol triclosan was dissolved in 100 mL tetrahydrofuran (THF). For acidic environment, 1 mL 0.1 M H_2SO_4 were added into 20 mL triclosan solution. The solution containing 0.1 g grafted fibers were spinned at 110 rpm for 12 h at 50 °C in a water bath with shaker. Fibers bound by triclosan were washed in water for 24 h and dried in an incubator at 50 °C.

2.6. FTIR spectrum

FTIR spectra of PET and vinyl monomer grafted PET fibers were obtained. The fibers were cut with scissors into roughly 1 mm size, mixed with KBr, and then pressed. Spectra were recorded with a Bruker Vertex 70V FTIR photometer.

2.7. Scanning electron microscopy (SEM) analysis

SEM analysis were performed employing JEOL Model JSM 5600 to analyze the surface morphology of the original and monomer grafted PET fibers coated with gold.

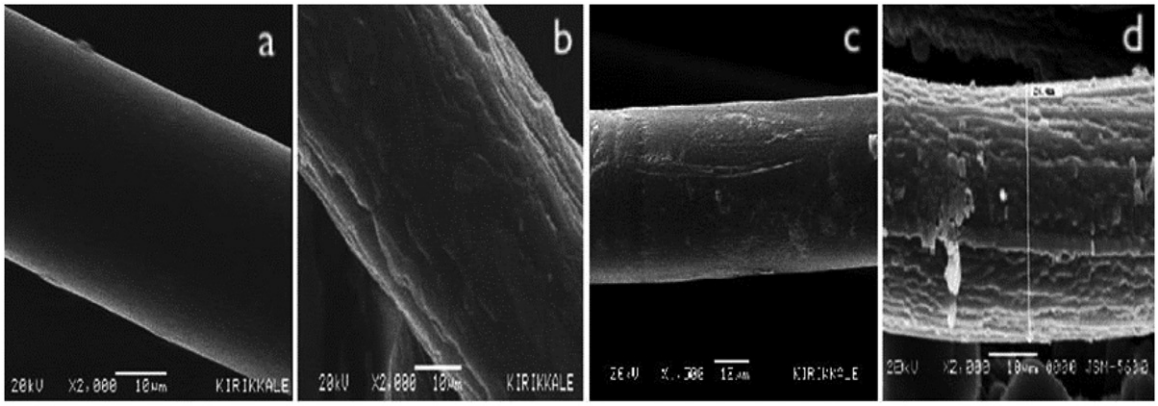

Figure 3. SEM images of (a) original fibers (b) 4-VP-g-PET fibers (c) MMA-g-PET fibers and (d) GMA-g-PET fibers.

2.8. Kirby-Bauer test

Sterile solid medium was poured into 90 mm and 200 mm petri dishes next to a bunsen burner. Bacteria culture with McFarland standard was plated with the streaking technique. Petri dishes plated with bacteria were incubated at room temperature for 5–6 min. Antibiotic disk, antibacteial pellet and empty discs were used as control groups (Figure 1). Petri dishes were incubated at 37 °C incubator for 18–24 h. Zone diameters were measured using a ruler[30].

2.9. Bacterial growth curve

Nutrient broth was used as liquid medium. 100 μl *S. aureus* sample from the bacteria culture was inoculated into 100 mL nutrient broth. In a total of six cultures, three control (without fibers) cultures and three cultures with the biggest zone diameters that have 0.1 g of fibers, were incubated on a shaker at 37 °C. The growth curve was drawn with the first measurement at time 0 and the other measurements at every 2 h using OD (optical density) at 600 nm with a spectrophotometer[31].

3. Results and discussion

This study aimed to give antibacterial properties to PET that was modified using graft copolymerization. Firstly, PET fibers were grafted with 4-VP[32], MMA[33], and GMA[34] (Figure 2). PET fibers grafted with 4-VP were modified with chlorine so that the chlorine was added into the fibers and oxidized (Figure 2(a)). MMA were first aminated and then modified with chlorine (Figure 2(b)). Moreover, fibers grafted with GMA and MMA were modified with triclosan that is known to have antibacterial properties (Figure 2(b), 2(c)). Characterization of the fibers gained antibacterial properties were examined by FTIR spectra and SEM images.

Scanning electron micrographs of original and 4-VP-g-PET fibers are shown in Figure 3. According to the results from SEM, smooth PET fibers surface (Figure 3(a)) has a straight and relatively homogenous morphology. Micro phases in the grafted 4-VP, MMA and GMA PET fibers and heterogenous morphology in the grafted copolymer (Figure 3(b), 3(c), 3(d)) can be shown as the proof of successful graft polymerization.

Characterization of the modified fibers was carried outby FT-IR analysis. FT-IR spectra of original, 4-VP-g-PET and N-oxide-4-VP-g-PET fibers were analyzed and shown in Figure 4(a). The FT-IR spectrum of original PET fibers were observed in C=O (1712 cm^{-1}), C=C and aliphatic C–H (1411 and 1578 cm^{-1}) PET fibers. The spectrum of 4-VP-g-PET changed after grafting with 4-VP. It was observed on the spectrum that a new peak was created at 1594 cm^{-1} and this was associated with the resonance peaks of the 4-VP groups. Upon N-oxidation of 4-VP-g-PET, the new peak was lost, and 4-VP ring was oxidized as a result of N-oxide-4-VP unit absorption.

The FT-IR spectra of the original, MMA grafted and functional amine group added fibers are shown in Figure 4(b). Upon MMA-g-PET fibers, a peak at 1720 cm^{-1} was observed due to COOH group of MMA. O–H tension of the carboxylic acids were observed in a wide range at 3600–2300 cm^{-1}. A new characteristic peak was observed at 1539 cm^{-1} due to the binding of N-H. At the aminated fibers spectrum, a peak was observed at 1627 cm^{-1} due to amide groups. Therefore, FT-IR results showed that the MMA monomers were grafted into PET fibers and amination took place successfully.

The changes in the fibers upon modification of MMA-g-PET fibers with triclosan is shown in Figure 4(c). A peak was observed at 1720 cm^{-1} due to COOH group of MMA. O–H tension of the carboxylic acids was observed in a wide range at 3600–2300 cm^{-1}. It was observed that the O–H peak due to COOH group of MMA at 3600 cm^{-1} was lost due to the addition of triclosan to the structure. Together with the addition of C–Cl bonds to the fibers structure a characteristic peak was observed at 576 cm^{-1}.

Chemical structures of the original, GMA-g-PET and Trc-GMA-g-PET fibers were analyzed with FT-IR spectroscopy and shown in Figure 4(d). FT-IR spectrum of the original PET fibers were observed and C=O (at 1712 cm^{-1}), C=C and aliphatic CH (at 1411 and 1578 cm^{-1}) peaks were seen. Upon graft polymerization with GMA, spectrum of GMA-g-PET has changed and a new peak was formed at 905 cm^{-1}. This peak resulted from the epoxy groups of GMA. Moreover, a characteristic peak of triclosan at 1247 cm^{-1} due to Ar-O-Ar and at 576 cm^{-1} due to C–Cl was observed.

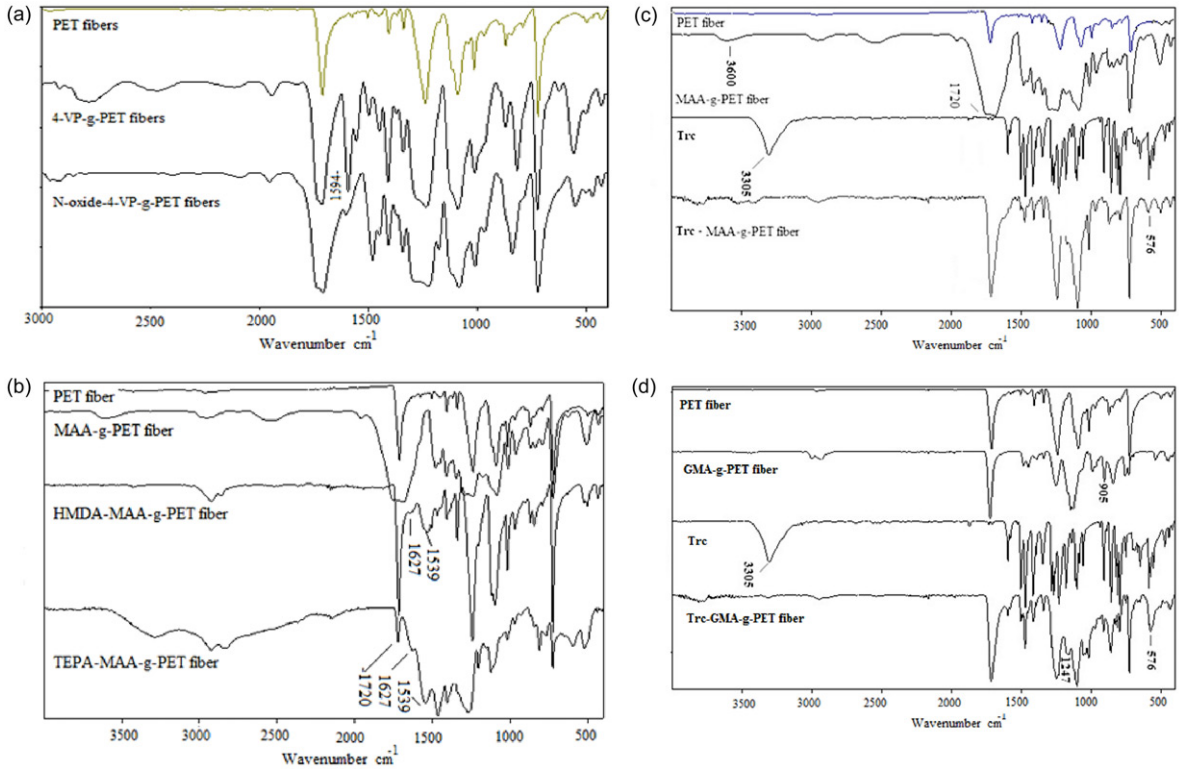

Figure 4. FT-IR analyses of (a) 4 -VP-g-PET fibers derivatives (b) aminated MMA-g-PET fibers (c) Trc-MMA-g-PET fibers (d) Trc-GMA-g-PET fibers.

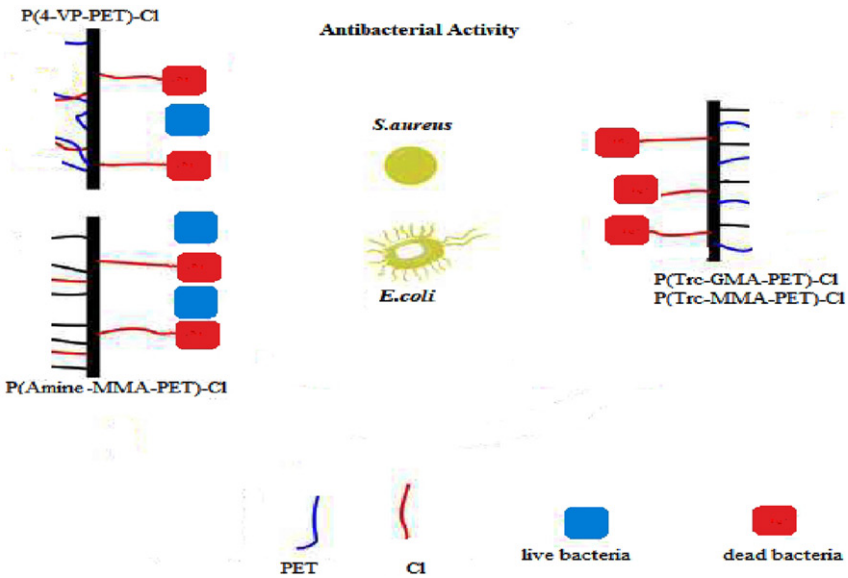

Figure 5. Antibacterial effect.

3.1. Kirby-Bauer test

Antibacterial PET fibers were prepared by the graft copolymerization of vinyl monomers (4-VP, MMA, GMA) and the modification of those with active functional groups (Cl, amine, Trc, N-oxide). Antibacterial effect of the PET fibers on bacteria (*E. coli* and *S. aureus*) is shown schematically in Figure 5. Antibacterial effect on bacteria was examined by the tests in solid and liquid media and the growth curve of the bacteria in the liquid medium was determined.

S. aureus and *E. Coli* plating was carried out by the streaking method. PET fibers and modified PET fibers are placed in disc form and antibacterial effects were compared. The antibacterial effect of modified PET fibers on *S. aureus* and *E. coli* was examined by the Kirby-Bauer Test. The antibacterial effect of the modified fibers was observed on both bacteria types in the medium by the observation of the inhibition zones (Figure 6) around the fibers discs. Inhibition diameters for *E.coli* and *S. aureus* are shown in Table 1. Accordingly, 13-mm-diameter discs were absorbed in the medium and the disc diameter after the multiplication in the medium was shown. Also, the zone diameters of the antibiotics are given in the table. When the zone diameters of the PET fibers and the

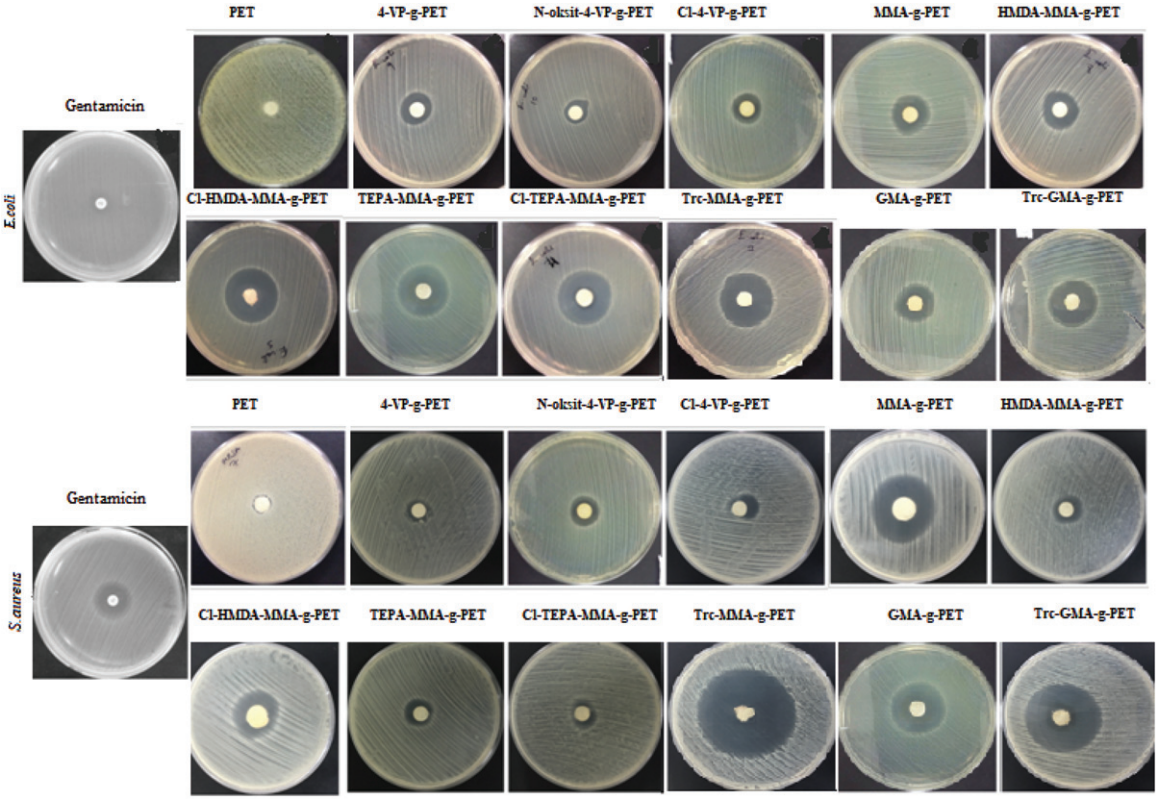

Figure 6. Kirby-Bauer test.

Table 1. The zone diameters of the PET fibers in the solid medium plated with *E. coli* and *S. aureus*.

Polymers	*E. coli* zone diameters (mm)	*S.aureus* zone diameters (mm)
Gentamicin (antibiotic)	15	26
Ungrafted PET fibers	0	0
4-VP-g-PET fibers	32	55
N-oxide-4-VP-g-PET fibers	28	32
Cl-4-VP-g-PET fibers	31	36
MMA-g-PET fibers	20	75
HMDA-MMA-g-PET fibers	28	30
Cl-HMDA-MMA-g-PET fibers	34	59
TEPA-MMA-g-PET fibers	26	31
Cl-TEPA-MMA-g-PET fibers	35	36
Trc-MMA-g-PET fibers	49	130
GMA-g-PET fibers	24	30
Trc-g-PET fibers	56	87

antibiotic was compared, antibiotic was observed to have a biggest inhibition zone in *S.aureus* than that in the fibers. When the disc diameters of the PET fibers were compared, the biggest inhibition diameter was observed in petri dish with *S. aureus* inoculation. Several compounds are synthesized and their antibacterial properties against *S. aureus* and *E. coli* are tested in the literature[35]. In a study, the antibacterial properties of the synthesized compounds against *E. coli* and *S. aureus* were tested and all the synthesized compounds were shown to be more effective on *S. aureus*[36].

3.2. Bacterial growth curve

The growth curve of *S. aureus* in the medium with and without Trc-MMA-g-PET fibers, which resulted in the biggest zone diameter, was determined and is shown in Figure 7. In

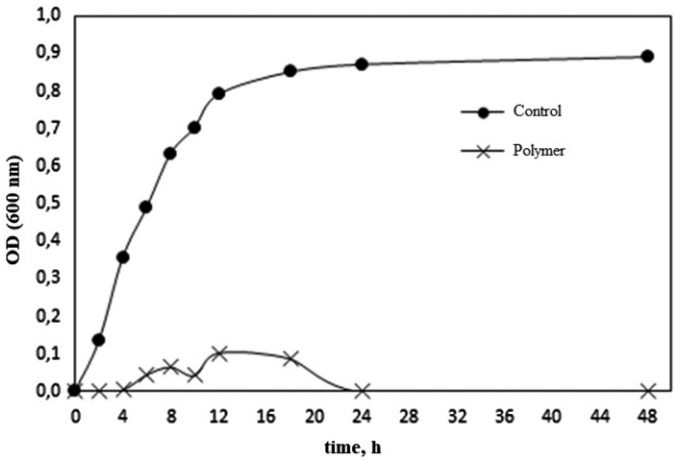

Figure 7. The growth curve of the Trc-MMA-g-PET fibers in the liquid medium.

the absence of the fibers, *S. aureus* proceeded to the logarithmic phase earlier whereas in the presence of the modified PET fibers, they could not reach the logarithmic phase and did not show any multiplication. According to the growth curve, PET fibers showed antibacterial properties, bacterial growth was induced, and antibacterial activity was increased.

4. Conclusions

4-VP, MMA, and GMA monomers were bound on the PET fibers successfully by the graft polymerization. Grafted PET fibers were modified in optimized conditions with several functional groups such as amine, chlorine, hydrogen peroxide, and triclosan to gain antibacterial feature. While the original PET fibers did not show antibacterial properties, the

modified fibers showed antibacterial properties against *S. aureus* and *E. coli*. Zone diameters formed in the solid medium was compared with the one formed upon antibiotic gentamicin use. *E.coli* zone diameter was 15 mm whereas *S. aureus* zone diameter was 26 mm. Zone diameters of the PET fibers formed in the solid medium was measured. As a result, PET fibers were observed to be the most effective on the Gram positive bacteria *S. aureus*. Examining, the zone diameters of the 4-VP PET fibers in the solid medium, antibacteial activity of the 4-VP alone was higher than its oxidized or chlorined forms. Antibacteial activity of MAA was observed in solid and liquid culture assays. It was observed that the activity of the aminated fibers was induced and their zone diameter increased when they were quartenized with chlorine. Fibers modified with triclosan had the biggest zone diameter. Furthermore, the pH of the environment of the triclosan changed its activity. Disc diffusion sensitivity test results were confirmed by the results from liquid culture test and the results were quantified. Polymer was found to have an antibacterial effect according to the growth curve of *S. aureus* inoculated and 0.1 g of Trc-MMA-g-PET fibers added liquid medium. As a result, it was shown that the potential of the polyester fibers PET can be improved by adding antibacterial properties to the PET fibers, which normally does not have any antibacterial property, with a cheap, easy, and fast modification in a for the environmental and public health.

Acknowledgements

Authors are grateful to the Kırıkkale University Research Funding for the financial support of this study.

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