



Preparation of active antibacterial LDPE surface through multistep physicochemical approach: I. Allylamine grafting, attachment of antibacterial agent and antibacterial activity assessment

František Bílek^a, Táňa Křížová^b, Marián Lehocký^{a,*}

^a Centre of Polymer Systems, Tomas Bata University in Zlín, Nam. T.G.M. 5555, 76001 Zlín, Czech Republic

^b Department of Food Analysis and Chemistry, Tomas Bata University in Zlín, Nam. T.G.M. 275, 76272 Zlín, Czech Republic

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ABSTRACT

Low-density polyethylene (LDPE) samples were treated in air plasma discharge, coated by polyallylamine brush through copolymeric grafting surface-from reaction and deposited four common antibacterial agents (benzalkonium chloride, bronopol, chlorhexidine and triclosan) to gain material with active antibacterial properties. Surface characteristics were evaluated by static contact angle measurement with surface energy evaluation ATR-FTIR, X-ray Photoelectron Spectroscopy (XPS) and SEM analysis. Inhibition zone on agar was used as *in vitro* test of antibacterial properties on two representative gram positive *Staphylococcus aureus* (*S. aureus*) and gram negative *Escherichia coli* (*E. coli*) strains. It was confirmed, that after grafting of polyallylamine, more antibacterial agent is immobilized on the surface. The highest increase of antibacterial activity was observed by the sample containing triclosan. Samples covered by bronopol did not show significant antibacterial activity.

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1. Introduction

Despite a great progress in the area of medical science, solving of problems referred to nosocomial infections are maintained at approximately the same level [1,2]. Nosocomial infections are diseases emerging during hospitalization and at once they are not related to the problem that the patient is hospitalized with. Such disorder brings worsening of health condition and patient comfort, extending the time of healing, additional antibiotic therapy, and therefore its price, the patient also becomes a source of infection for his environment and these complications frequently cause patient death [3]. Nosocomial infections may be endogenous or exogenous origin. Infections of endogenous (internal) origin are caused by microorganism normally occurred in the human body, that is weakened during immunodepression. For exogenous (external) source of nosocomial infections lies a factor in the surroundings: other patients, hospital staff, insects, air, food, but highly potential source of infection are foreign objects introduced into the patient body as medical devices. These are for example: urinary catheters, central vascular cannulae, tubes of respirators or artificial prostheses [4]. The surface of such devices can be easily colonized by bacteria, when hardly removable biofilm is created as a source and

centre of further infection [5]. This infection may be partly reduced by compliance with hygienic-epidemiological regime of nursing staff. Equally important way how to reduce foreign object infection risk on in human body on minimum is to treat its surface to gain active antibacterial properties [6].

Materials with antibacterial properties can be prepared by several approaches [7–9]. One of them is immobilization of antibacterial agent on the surface of polymer material [10]. This seems to be more effective than incorporation of the active species into the polymer bulk with respect to the relatively short application period of these devices which is mostly not extending 2 weeks. Surface modification of synthetic polymer materials changes the chemical composition of the upper layer [11–13]. The overall substrate properties, especially mechanical, remain unchanged and the substrate achieves adequate strength and flexibility [6,14].

Surface modification can be achieved by several methods, i.e.: mechanical treatment, flame treatment, wet chemical etching by strong oxidizing acids, corona or plasma treatment [15–17]. The last listed method seems to be the most effective as well as environmental friendly. Plasma treatment of polymer materials in air as a carrier gas is an effective tool to modify the surface via incorporation of oxide containing groups onto the surface structure, i.e.: hydroxyl, carbonyl, carboxyl, ether, hydroperoxide, etc. Some of the groups are unstable and system leads to the reaction forming active radicals [18–20]. These metastables are capable to react with suitable monomer creating polymer “brush-like” structure

* Corresponding author. Tel.: +420 608616048.

E-mail address: lehocky@post.cz (M. Lehocký).

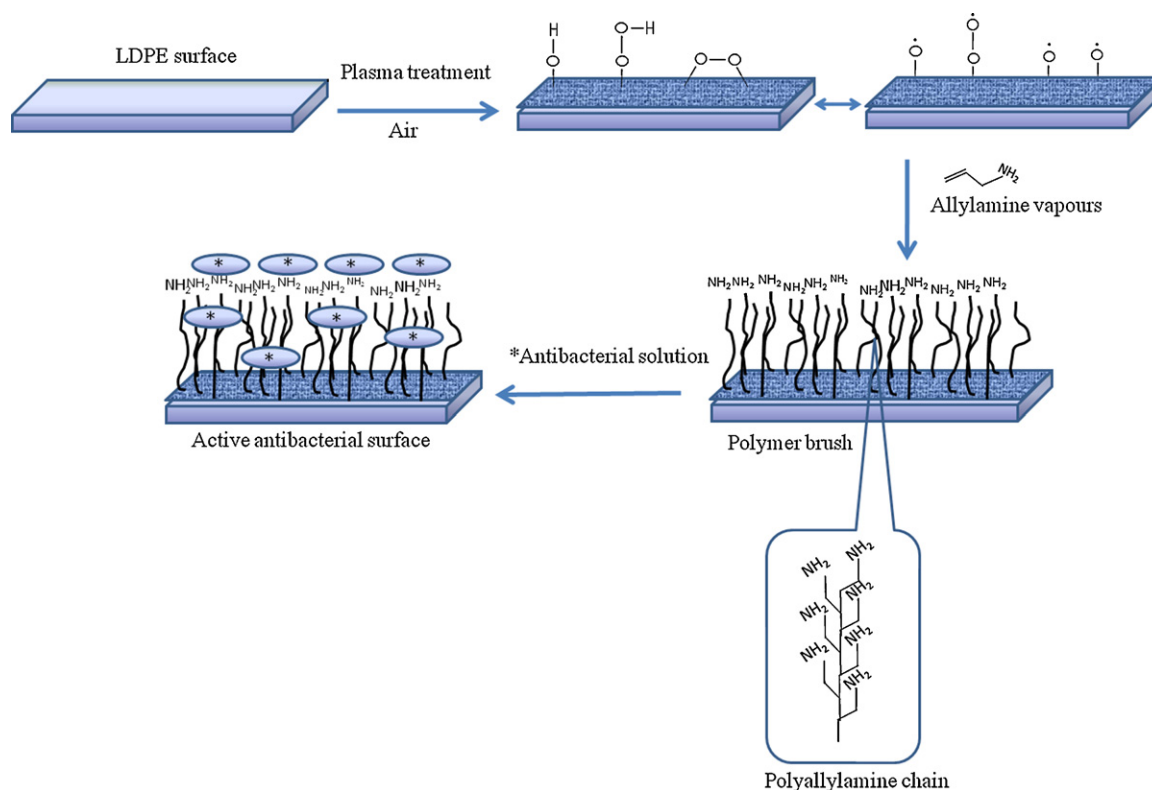


Fig. 1. Modification of LDPE surface.

[21–23]. This kind of structure is suitable for reaction with antibacterial agent molecule functionalities by creation of intra-molecular forces. It is worth noting that chemical bond due to its relative high strength would not lead to final application because of the relatively low antibacterial agent diffusion from the surface structure and thus low active antibacterial activity [24–26].

In this work, the radiofrequency (RF) plasma discharge was selected for surface activation of low-density polyethylene (LDPE) substrates as a first operation in the multistep approach. After surface modification the allylamine (AAM) monomer was grafted onto the surface to create amine containing polymer brush structure via radical “surface from” reaction in gaseous phase. The last step is connected with the anchoring of the active antibacterial species into the surface structure.

Benzalkonium chloride, bronopol, chlorhexidine and triclosan were chosen as common antibacterial substrates in our study with respect to the final application which does not allow utilization of potentially hazardous antibacterial chemical reagents which cannot be used in direct contact with the body [6,27–29].

In this paper, the active antibacterial material multistep preparation approach, surface characterization by contact angle measurements, ATR-TIR spectroscopy, Scanning electron microscopy, X-ray Photoelectron Spectroscopy (XPS) and antibacterial assay is described in order to prepare a novel active functional material which can bring a new potential in healthcare reducing the amount of nosocomial infections and therefore to improve the comfort of the patients amongst others. These results can be useful not only in the development of packaging or medical material, but can contribute to the further examination of phenomena happening between polymer surface and bacteria cell.

2. Methods

The LDPE film samples of the 0.1 mm thickness with square size of 5 cm × 5 cm were washed in solution of cationactive ten-

side, cleaned in water and consequently in deionized water. After cleaning, they were dried in laboratory conditions for 2 h. Such prepared samples were subjected to RF plasma treatment from both sides by using FEMTO Diener (Germany) plasma reactor. The discharge was operated at the frequency of 13.56 MHz at the capacitively coupled radiofrequency reactor. The discharge matching power was 50 W for all experiments. For all samples, 5 min was the duration of plasma process. Flow rate of air as a carrier gas was 50 sccm. After that, the samples were placed in allylamine (Fluka) vapours for 15 s in order to create the amine polymer brush. Then, the samples were separately rinsed into the reaction vials containing antibacterial agent solutions and kept standing for 24 h at laboratory conditions. Concentration of antibacterial solution were following: 2% (w/v) Triclosan (TC) in absolute ethanol, 2% (w/v) bronopol (BrP) in absolute ethanol, 2% (w/v) benzalkonium chloride (BCh) aq. solution, 2% (w/v) chlorhexidine (ChlH) in isopropylalcohol 70% (v/v) aq. solution. Finally, the samples were taken out of the solution and cleaned by stream of water followed by ultrasonication for 5 min in deionized water to eliminate the unanchored antibacterial agent from sample surface. The last cleaning was done by deionized water. Cleaned samples were dried in ambient conditions for 2 h and then separately placed in desiccator.

2.1. Reagents

LDPE polymer was obtained as packing foil (The Dow Chemical Company), Triclosan ([5-chloro-2-(2,4-dichlorophenoxy) phenol]) 97.0%, bronopol (2-bromo-2-nitropropane-1,3-diol), chlorhexidine (1,1-hexamethylene bis[5-(4-chlorophenyl)biguanide]), benzalkonium chloride, absolute ethanol and allylamine were supplied by Sigma Aldrich (USA). Isopropylalcohol 99.7% was gained from Lach-Ner (Czech Republic). All reagents were used as received without further purification.

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