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ORIGINAL ARTICLE

Synthesis and biocidal activity of modified poly(vinyl alcohol)

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Abstract Functionalized polymers and their polymer nature give them more advantages than the corresponding small molecules. In this respect, polymeric ammonium and phosphonium salts were prepared by chemical modifications of poly(vinyl alcohol) (PVA) aiming to explore their antimicrobial activities against pathogenic bacteria and fungi. The modifications were performed by chloroacetylation with chloroacetyl chloride. Incorporation of the ammonium and phosphonium salts was conducted by the reaction of chloroacetylated poly(vinyl alcohol) (CPVA) with triethylamine (TEA), triphenylphosphine (TPP), and tributylphosphine (TBP). The antimicrobial activity of the polymers against variety of test microorganisms was examined by the cut plug and viable cell counting methods of shake cultures of 10 times dilute nutrient broth and Sabouraud's media, seeded with the test microorganisms. It was found that the immobilized polymers exhibited antimicrobial activity against the Gram negative bacteria (*Escherichia coli, Pseudomonas aeruginosa, Shigella* sp. *and Salmonella typhi*) and Gram positive bacteria (*Bacillus subtilis and B. cereus*) and the dermatophyte fungus (*Trichophyton rubrum*). The growth inhibition of the test microorganisms (ratio of surviving cell number, M/C) varied according to the composition of the active group in the polymer and the test organism. It increased by increasing the concentration of the polymer. Triphenyl phosphonium

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salt of the modified poly(vinyl alcohol) exhibited the most biocidal activity against both Gram-negative and Gram-positive bacteria after 24 h.

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1. Introduction

Recently, much attention has been paid to the problems of environmental pollution and health. Infection by pathogenic microorganisms is of great concern in many fields, particularly in medical devices, drugs, health care products and hygienic applications, water purification systems, hospital and hospital furniture, dental surgery equipment, textiles, food packaging, food storage, etc. (Kenawy et al., 2007a, 2009)

Antimicrobial agents are used for many applications, such as water sterilization, drugs, soil sterilization, biomedical-device sterilization, and prevention of the microbial contamination of shipboard, water compensated, and hydration fuel tanks (Kenawy et al., 1998, 2002, 2005, 2006, 2011). They are also in common use in the areas of health care and hygienic applications such as sterile bandages and clothing (e.g., antimicrobial surgical gowns and antifungal athletic socks). Furthermore, antimicrobial agents are commonly used in coating of surfaces such as ship hulls, shower walls, and many kinds of tubing to minimize the problems of biofouling (Kenawy et al., 2006; Worley and Sun, 1996). The use of antimicrobial polymers offers promise for enhancing the efficacy of some existing antimicrobial agents and minimizing the environmental problems accompanying conventional antimicrobial agents by reducing the residual toxicity of the agents, increasing their efficiency and selectivity, and prolonging the lifetime of the antimicrobial agents. Also, polymeric antimicrobial agents have the advantage that they are nonvolatile and chemically stable and do not permeate through skin. Therefore, they can reduce losses associated with volatilization, photolytic decomposition, and transportation. In the field of biomedical polymers, infections associated with biomaterials represent a significant challenge to the more widespread application of medical implants (Kenawy et al., 2007b; Acharya et al., 2005; Jiang et al., 2004).

Considerable interest has been focused on functional polymers and on their diverse applications in many fields such as biomedical applications including drug delivery and antimicrobial polymers. Functional polymers have the potential advantages over small analogous molecules. Their usefulness is related to both the functional groups and to their polymeric nature whose characteristic properties depend on the extraordinarily large size of the molecules (El-Newehy et al., 2012b; Kenawy et al., 1998; Akelah and Moet, 1990).

Poly(vinyl alcohol) (PVA) is a hydrophilic polymer, has unique properties. It absorbs water and swells easily, but the swelling is inhibited by salts. Its physico-chemical properties depend on the degree of poly(vinyl acetate) hydrolysis. The solubility of PVA in water increases greatly as the degree of acetate group hydrolysis increases. Moreover, PVA can be used for releasing biological and medical materials in a controlled way (Kenawy et al., 2007; Zhang et al., 2005; Gimenez et al., 1996). PVA was used in some modern technologies, such as hydrogels, polyelectrolytes, optics, and biomaterials including soft contact lenses, implants, and artificial organs. This is due to their inherent non-toxicity, non-carcinogenicity, good biocompatibility, and high degree of swelling in aqueous solutions. The PVA hydrogels were used as drug delivery matrices or in the form of powders added to mixtures of other excipients for tablet formation (Kenawy et al., 2007; Zhang et al., 2005; Gimenez et al., 1996; Colombo et al., 1985; Carstensen et al., 1981). In our previous work, we have reported the use of PVA as a controlled release device for drugs using the freezing and thawing technique (Kenawy et al., 2010) and the electrospinning technique (El-Newehy and Alamri, 2012a; Kenawy et al., 2007)

In this study, we prepared immobilized polycationic biocides by modification of poly(vinyl alcohol) via chemical modification with chloroacetyl chloride followed by immobilization of onium groups onto the chloroacetylated polymer. Antimicrobial activities of the resultant polymers were explored *in vitro* by cut plug and the viable cell counting methods against strains of appropriate microorganisms.

2. Experimental

2.1. Materials and instruments

Poly(vinyl alcohol) (PVA) (M_w 13k–23k, 98% hydrolyzed), tributylphosphine (TBP), triphenylphosphine (TPP), and chloroacetyl chloride were purchased from Aldrich. Triethylamine (TEA) was of purest grade available from Merck-Schuchardt and was used without further purification. Pyridine was dried before use. All solvents were dried and distilled before use.

IR Spectroscopy: IR spectra were recorded on a PERKIN–ELMER 1430 Ratio Recording Infrared Spectrophotometer from KBr pellets.

Elemental microanalyses were determined on Heraeus (Microanalysis Center, Cairo University, Egypt) and Elemental Analyzer Mode 1106 Carlo Erba Strumentazione (Pisa University, Pisa, Italy).

2.2. Test microorganisms and media used

The test microorganisms included were Gram negative bacteria; *Escherichia coli, Pseudomonas aeruginosa, Shigella sp.* and *Salmonella typhi*, and the Gram positive bacteria; *Bacillus subtilis* and *B. cereus*, in addition to the dermatophyte fungus *Trichophyton*. The test microorganisms were obtained from the culture collection of the Bacteriology Unit, Botany Department, Faculty of Science, Tanta University, Egypt. Nutrient and Sabouraud's broths and nutrient and Sabouraud's agar were used for growing and maintenance of the test bacteria and fungi.

2.3. Chloroacetylation of poly(vinyl alcohol)

In a two-neck round-bottomed flask, pyridine (55 mL, 681.8 mmol) was added to a suspension of PVA (I) (10 g, 227.3 mmol) in chloroform and the mixture was cooled to 0 °C in an ice-salt bath. To the cold mixture, chloroacetyl chloride (55 mL, 681.8 mmol) was added dropwise with vigorous

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