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Growth of ZnO nanowires on polypropylene membrane surface—Characterization and reactivity

Marta Bojarska[a,b,*], Bartosz Nowak[a], Jarosław Skowroński[c], Wojciech Piątkiewicz[c,d], Leon Gradoń[a]

[a] Warsaw University of Technology, Faculty of Chemical and Process Engineering, Waryńskiego 1, 00-645 Warsaw, Poland
[b] Lehrstuhl für Technische Chemie II, Universität Duisburg-Essen, Essen 45117, Germany
[c] Institute for Sustainable Technologies—National Research Institute, Pułaskiego 6/10, 26-600 Radom, Poland
[d] PolymemTech Sp. z o.o., al. Niepodległości 118/90, 02-577 Warsaw, Poland

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ABSTRACT

Need for a new membrane is clearly visible in recent studies, mostly due to the fouling phenomenon. Authors, focused on problem of biofouling caused by microorganisms that are present in water environment. An attempt to form a new membrane with zinc oxide (ZnO) nanowires was made; where plasma treatment was used as a first step of modification followed by chemical bath deposition. Such membrane will exhibit additional reactive properties. ZnO, because of its antibacterial and photocatalytic properties, is more and more often used in commercial applications. The authors used SEM imaging, measurement of the contact angle, XRD and the FT–IR analysis for membrane characterization. Amount of ZnO deposited on membrane surface was also investigated by dithizone method. Photocatalytic properties of such membranes were examined through methylene blue and humic acid degradation in laboratory scale modules with LEDs as either: wide range white or UV light source. Antibacterial and antifouling properties of polypropylene membranes modified with ZnO nanowires were examined through a series of tests involving microorganisms: model gram-positive and –negative bacteria. The obtained results showed that it is possible to modify the membrane surface in such a way, that additional reactive properties will be given. Thus, not only did the membrane become a physical barrier, but also turned out to be a reactive one.

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1. Introduction

With the increasing world population, problems concerning water pollution grew significantly in last few years and that is why the use of disinfectants is so important [1–3]. Pathogenic microorganisms such as: *Staphylococcus, Enterococcus,* or *Streptococcus,* present in natural water and wastewaters, are a serious threat to public health [4,5]. Membranes can be used for: bacteria, macromolecules, organic compounds, salts or even viruses removal. In the last couple of years membranes become more widespread because of their reliability and ease of use [2,6–9]. Particularly, the microfiltration turned out to be less costly than other purification methods. Microfiltration is often used as a pre–filtration process prior to nanofiltration, reverse osmosis, or as a key element in bioreactors [6,8]. Despite significant potential of microfiltration and membranes, problem of organic fouling and biofouling lowers their efficiency [4,6,7]. Also large amounts of natural organic matter (*NOM*) in e.g. surface waters make organic fouling inevitable. Fouling is mostly caused by formation of a filtration cake on the membrane surface, which blocks pores or reduce their size. [10,11]. Biofouling or microbiological fouling is considered to be far worse because of microorganisms ability to multiply and surface colonization [6–8]. The moment the microorganism sticks to the surface a complicated, multistage process leading to biofilm formation begins. At the same time extracellular polymeric substances (EPS) such as polysaccharides and other organic substances (e.g. proteins

Abbreviation: CBD, chemical bath deposition; CFU, colony forming units; CIP, cleaning in place; COD, chemical oxygen demand; EPS, extracellular polymeric substances; HA, humic acid; HMTA, hexamethylenetetramine; HT, hydrothermal growth; LB, Lysogeny Broth; M9, minimal broth; MB, methylene blue; NOM, natural organic matter; OD, optical density; ROS, reactive oxygen species.

* Corresponding author at: Warsaw University of Technology, Faculty of Chemical and Process Engineering, Waryńskiego 1, 00-645 Warsaw, Poland.
E-mail addresses: m.bojarska@ichip.pw.edu.pl (M. Bojarska), novakbartosz@gmail.com (B. Nowak), jaroslaw.skowronski@itee.radom.pl (J. Skowroński), w.piatkiewicz@polymemtech.com (W. Piątkiewicz), l.gradon@ichip.pw.edu.pl (L. Gradoń).

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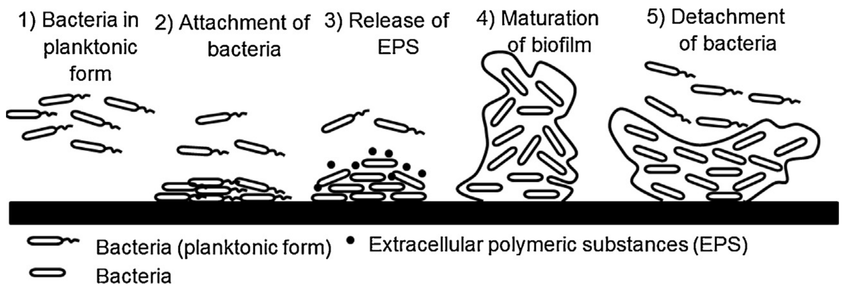

Fig. 1. Scheme presenting stages of biofilm formation.

or nucleic acids) are released [7,12]. Biofilm formation presented in Fig. 1 [6,11–13] includes the following stages:

- bacteria in planktonic form (1);
- attachment of bacteria (2);
- release of EPS (3);
- maturation of biofilm (4);
- detachment (release) of bacteria (5).

In case of membrane filtration there are several ways to reduce the problem of fouling/biofouling, like: cross–flow filtration, stream disinfection (pasteurization, UV radiation, potassium permanganate, chlorates, etc.), back puls, or cleaning in place (CIP) [10]. Total recovery of the filtration stream is impossible due to irreversible adhesion of some biofilm components to the membrane surface. First stages of biofilm formation require a membrane–pollutant interaction, thus it is crucial to interrupt this process at this stage. Membrane surface modification can reduce the problem of fouling [6,9,10,12,14,15]. That is why there is a need for new membranes, with photocatalytic or antibacterial properties. Those properties may reduce the problem of both organic fouling and biofouling [4]. There are two types of membranes with nanoparticles in their structure: membranes with a thin layer of nanoparticles on their surface or mix matrix membranes, which have nanoparticles in all their volume [16]. Due to surface nature of the fouling phenomena, authors decided on the first approach.

ZnO is common in natural world and it is environmentally friendly [17–23]. Zinc oxide is a semiconductor with of wide band gap of 3.37 eV and high bonding energy (60 meV). ZnO can absorb UV radiation with the wavelength not exceeding 385 nm, which is connected with band gap, and visible light with the peak between 450 and 750 nm, which can be associated with the defect in the zinc oxide crystal [23]. In case of this research, absorption peaks related to crystal defects are desired. Jones et al. [5] reported in their work that 4% or less UV radiation present in fluorescent light is sufficient for the activation of zinc oxide. In their work they also claim that visible light, present in the laboratory, is sufficient for activation of ZnO [5]. Because of high electron mobility (better than TiO_2) and ease of fabrication, ZnO-based nanostructures are of interest to the photocatalysis [24]. ZnO doping by Graphene Oxide [25], Sn [26], Ag [27] or Ni [28], can even enhance photocatalytic activity of ZnO in visible light, or reduce the e^-–h^+ pair recombination. Thanks to its photocatalytic properties, zinc oxide can be used for prevention of organic fouling. Photocatalysis is process activated by light, where on the semiconductor an electron–hole pair is generated (Fig. 2) [21,29,30].

Pair e^-–h^+ participates in redox reactions with elements adsorbed on the surface of catalyst. The hole can oxidize water to a hydroxyl radical, which then can initiate reactions leading to oxidation of organic compounds. Electron can be transferred to an electron acceptor, e.g. oxygen or metal ion, which has more positive redox potential than the band gap of the photocatalyst (reduction of metal ion to metal and deposition on photocatalyst surface) [31]. Photocatalysis is used for decolorization, degradation of dyes, chemical oxygen demand (COD) reduction, mineralization of toxic organic components, removal of heavy metal ions, degradation of harmful fungicides, herbicides, pesticides, water purification and disinfection [29,31,32].

Zinc oxide also exhibits high toxic properties against both gram–positive and –negative bacteria [2,33–37]. ZnO can also destroy spores [38]. It exhibits anti–fungal properties, for which it has been known since the 1990s, e.g. against *Aspergillus brasiliensis*—aggressive and widely occurring fungus [36,39]. Mechanism of antibacterial activity of zinc oxide is not well known. It is assumed, that it is mainly based on the formation of reactive oxygen species (ROS) from water and oxygen that disrupt the integrity of bacterial membrane, oxidize proteins and damage DNA or inhibit replication of DNA. The role of zinc ions is not clear, it is suggested that ions bind with the membrane of microorganisms, and extend the lag phase during the growth of bacteria; they also can damage DNA, disrupt enzymes, interrupt transmembrane electron transport and cause protein denaturation (Fig. 3) [2,33,36,37,39,40,41]. In addition, microorganisms that have been deposited on wurtzite modified membrane surface may be mechanically damaged by the nanowires. The cell is suspended on nanowires and the cell wall breaks due to the resulting mechanical stress [28,42]. Authors focused on ZnO as the modification material, due to described above few antibacterial mechanisms. In contrast, more widely used titanium dioxide antibacterial mechanism, is mainly associated with ROS formation and oxidative stress [43].

Zinc oxide nanoparticles are toxic to e.g.: *Escherichia coli* [35,37,39], *Bacillus subtilis* [35,37,39], *Pseudomonas fluorescens* [39], *Pseudomonas aeruginosa* [37], *Staphylococcus epidermis* [5,37], *Staphylococcus agalactiae* [39], *Staphylococcus aureus* [5,37,39,44], *Candida albican* [37], *Enterococcus faecalis* [37], *Streptococcus pyogenes* [5] or *Streptococcus mutans* [45]. There are also studies on the ZnO toxicity with respect to their potential environmental effects (influence of ZnO on algae—*Chlorella* sp.) [46]. Still, there is no clear

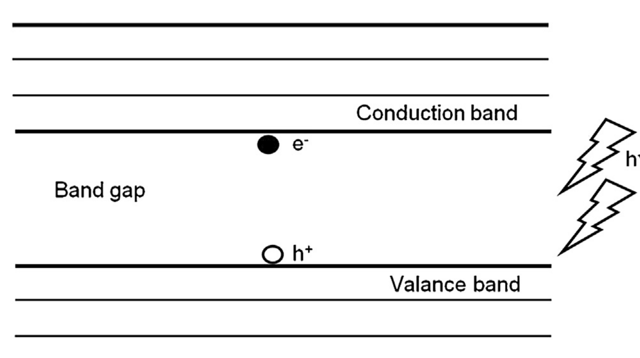

Fig. 2. Scheme of the hole (h^+) and electron (e^-) pair formation on the surface of the semiconductor.

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