Journal of Machine
Construction
and Maintenance
QUARTERLY 1/2019(112)
ISSN 1232-9312

p. 69–76

Andrzej MAJCHER*, Anna KOWALIK-KLIMCZAK
Institute for Sustainable Technologies – National Research Institute, Radom, Poland
* Corresponding author: andrzej.majcher@itee.radom.pl

ULTRASONIC IMPLEMENTATION OF NANOPARTICLES ON POLYMERIC FILTRATION MATERIALS

Key words: membrane, filter, biofouling, antibacterial properties, ultrasounds, coated nanoparticles.

Abstract: Biofouling is a phenomenon that adversely affects the efficiency of filtration hindering wide the application of filtration materials. One of the methods for biofouling prevention is coating of antibacterial nanoparticles on a surface of filters and membranes using ultrasound. In the paper, the possibility of using an ultrasonic method was evaluated to implement nanoparticles of titanium dioxide (TiO_2) and zinc oxide (ZnO) on polymeric filtration materials via different types of transducers and selected parameters of the ultrasound generator. Ultrasonic methods of modification were found to be efficient in terms of imparting antibacterial properties; however, the functional additives coated on polymeric materials were characterized by poor resistance to leaching. Thus, further work needs to be performed to improve the adhesion and stability of nanoparticles onto polymeric materials to enable the generation of filtration materials characterized by high stability during real operating conditions.

Ultradźwiękowa implementacja nanocząstek na polimerowych materiałach filtracyjnych

Słowa kluczowe: membrana, filtr, biofouling, właściwości antybakteryjne, ultradźwięki, nanoszenie nanocząstek.

Streszczenie: Biofouling materiałów filtracyjnych jest zjawiskiem niekorzystnie wpływającym na efektywność filtracji i hamującym wprowadzanie tej technologii w nowe obszary zastosowań. Jedną z metod zapobiegania biofoulingowi jest modyfikacja powierzchni membran antybakteryjnymi nanocząstkami. W artykule opisano prace studialne nad wykorzystaniem do tego celu metody nanoszenia ultradźwiękowego. Przedstawiono konstrukcje i parametry zestawionych stanowisk do generacji ultradźwięków, wyniki procesów nanoszenia nanocząstek tlenku tytanu i tlenku cynku, wpływ głównych parametrów procesu na strukturę uzyskiwanych pokryć. Oceniono ich właściwości antybakteryjne oraz odporność na wymywanie. Określono kierunki dalszych prac mających na celu wytworzenie metodą ultradźwiękową materiałów filtracyjnych charakteryzujących się stabilnością podczas pracy w warunkach rzeczywistych.

Introduction

The biggest problem associated with filtration in both *dead-end* and *cross-flow* systems is biofouling created by settling forms of microorganisms on the surface and in the structure of filtration materials resulting in a rapid drop of filtration efficiency, increased operating costs, and adverse impacts on the environment when using biocides and chemical agents to clean filtration plants [1]. The nutrients contained in the liquid to be filtered promote microorganisms to grow and facilitates forming a biofilm on polymeric materials. The growth of biofilm on the surface of filtration materials limits the wide use of filtration, particularly in the recovery of drinking water from saline water and wastewater as well as in the closing of water cycles in industry. Therefore, the approaches that can effectively reduce biofouling have been extensively sought [2–6]. The most commonly known modification methods include the following:

- Material (e.g., new membrane materials, membrane modifications, membrane morphology shaping);
- Physical (e.g., aeration, dedicated membrane constructions, hydrodynamic methods, methods using an electromagnetic field);
- Chemical (e.g., chemical cleaning, chemical additives);

- Operational (e.g. additional stirring of the suspension, maintenance of the critical stream, periodic backwashing, interrupted filtration cycles); and,
- Biological (e.g., enzymes deactivation, energy decoupling, quorum quenching, cell wall hydrolysis, and bacteriophages).

However, these methods have significant limitations resulting from negative impacts on the efficiency and costs of filtration [4]. Innovative solutions that create great perspective for preventing biofouling are sonic implementation methods of bioactive nanoparticles on the surface of filtration materials. These methods use the phenomenon of the formation and implosion of microscopic cavitation bubbles in the wall layer of a colloidal suspension of nanoparticles (ultrasonic method) or their precursors (sonochemical method). Cavitation microbubbles arise as a result of the absorption of ultrasound energy provided by properly constructed and controlled sonic generators of relatively high power. In the centre of the collapsing bubble, a high pressure is created accelerating the nanoparticles towards the covered object, causing them to "stick" into its surface. This method has already been widely used to deposit metal nanoparticles and metal oxides on the surface of various types of utility materials, particularly to fabrics, nylon, and polyester [7, 8]. It was found that silver chloride nanoparticles can be used on silk yarn [9], and zinc oxide nanoparticles [10], copper oxide [11] and titanium dioxide [12] can be used to modify cotton fabrics.

There are few examples described in the literature on the application of a sonochemical coating with nanoparticles to polymeric filtration materials. Beyon and Kim [18] obtained Teflon membranes sonochemically coated with copper nanoparticles, which were used as catalyst carriers in a steam reforming processes of methanol at 22°C and 42°C. In turn, Svirinovsky et al. [19] obtained membranes with a two-layer structure on a partially fluorinated thermoplastic polymer, where the first layer contained ZnO or CuO nanoparticles and the second layer was a polymeric substrate. In contrast, typical technologies used to modify the surface of filtration materials are dip-coating [14], mixing the polymer in a solution of nanoparticles [15], the CVD (Chemical Vapor Deposition) method [16], and the PVD (Physical Vapour Deposition) magnetron sputtering technique [17].

The aim of the study was to assess the possibility of using ultrasounds generated using various types of transducers to implement nanoparticles on the surfaces of polymeric filtration materials.

1. Materials and methods

The choice of materials and methods was focused on ensuring the feasibility of experiments in the widest possible range of process parameters and the scalability of the results obtained for industrial applications. In this work, two types of nanoparticles, two types of ultrasound generation systems, and the standard method of testing antibacterial properties and resistance to leaching were used.

1.1. Materials

- Suspension of titanium dioxide nanoparticles (TiO_2) and suspension of 0.1% wt zinc oxide (ZnO) nanoparticles were prepared by mixing 35 nm nanoparticles (Nanografi) or 32 nm TiO_2 nanoparticles (anatase, Alfa Aesar) with demineralized water.
- Magna Nylon filter membranes with a diameter of 47 mm and a pore size of 0.22 μm (GVS Filter Technology) and SupaSpun II depth filters with a porosity of 1 μm (Amazon Filters) were used.

1.2. Ultrasound system

1.2.1. Stand with plate transducers (PP)

The stand was equipped with two plate transducers (Fig. 1) of 150 W, which were placed in a reaction tank with dimensions of 300×150×150 mm (l, w, h).

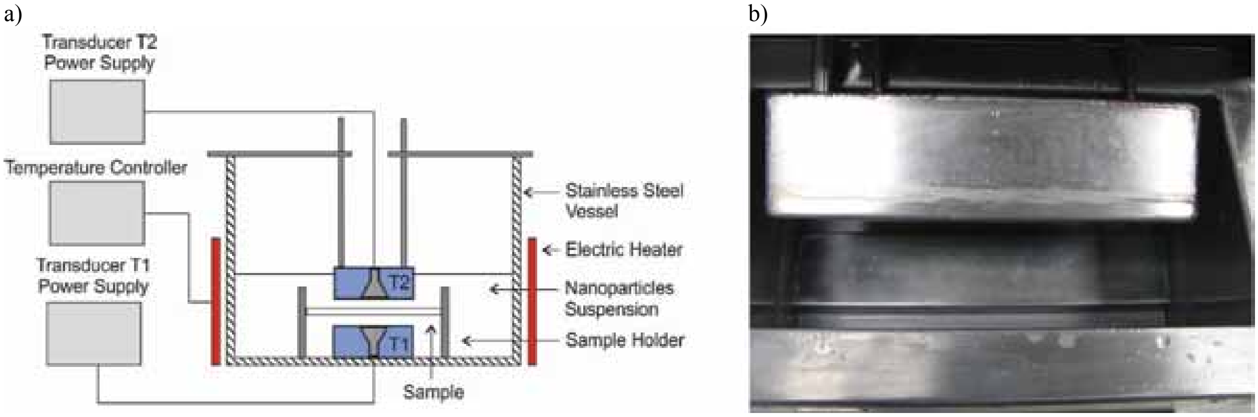


Fig. 1. Stand with plate transducers (a) and the interior of reaction chamber with plate transducers T1 and T2 (b)

The plate transducers (InterSonic, Poland) contain a set of single transducers hermetically sealed and attached to a common plate (Fig. 1b). The dimensions of the plates (60x250 mm) and the regulation of the power supply of the transducer allow obtaining the regulated intensity of ultrasounds up to 2 Wcm^{-2}. The suspension temperature was controlled by electric heaters, and the temperature was measurement with a PT100 thermocouple.

1.2.2. Stand with tube transducer (PT)

The second stand was equipped with a tube transducer (Fig. 2) with a power of 250 W and dimensions ϕ 30 mm, l 230 mm (InterSonic, Poland), placed horizontally in the reaction chamber with dimensions 400×140×200mm (l, w, h), in 20 mm from its bottom. Assuming an even distribution of ultrasounds on the surface of the transducer, the maximum intensity of ultrasounds was 1.03 Wcm^{-2}.

The temperature of the suspension was controlled using a H30/30D heating plate (CAT) under the reaction chamber. The temperature is measured with a PT100 thermocouple on the surface of the plate and in the suspension avoiding over-regulation of the temperature.

1.2.3. Pomiar częstotliwości, natężenia ultradźwięków i kawitacji

The OPCAV type meter (PBP Optel, Poland) with a measurement range of 20–50 kHz for ultrasounds and 0.4–1.2 MHz for cavitation was used to measure ultrasounds intensity and cavitation. The frequency of the generated ultrasound was measured by direct connection of the OPCAV meter probe used as a piezoelectric ultrasonic microphone to a digital oscilloscope type MSO 4054 (Tektronix) with a band of 500 MHz and a sampling rate of 2.5 GS/s.

1.2.4. Characteristics of the obtained layers

For imaging of polymeric filtration materials after coating with nanoparticles, a scanning electron microscope with SU-70 field emission type (Hitachi) and digital VHX-6000 Digital Microscope (Keyence Corporation) were used. The composition of elements deposited on materials was analysed using an Energy Dispersive X-ray Spectrometer (EDS) (Thermo Scientific).

1.2.5. Antibacterial properties

The antibacterial properties of the polymeric filters were evaluated under dynamic contact conditions in accordance with ASTM E2149 (Standard Test Method for Determining the Antimicrobial Agents Under Dynamic Contact Conditions). The ASTM E2149-13a method consists in shaking the appropriate mass of material for a limited time in the presence of the recommended bacterial strain (*Escherichia coli*, ATCC 25922) and a set of reagents. This method is a sensitive test, which is often used to measure the antimicrobial activity of irregularly shaped and/or hydrophobic materials. The antibacterial properties of materials depend on the direct contact of microorganisms with an active chemical component. The modified materials were shaken in a bacterial suspension of $1.5–3.0 \cdot 10^5$ CFU/mL for 1 h. The suspension was then diluted before and after contact with the material and spread on agar LB (liquid broth) plates. The plates were incubated for 24 h at 37°C, followed by the counting of colony forming units (CFU). The results were expressed as a percentage reduction in the viability of the bacterial cells.

To assess the antibacterial properties of membranes, a method developed at the Industrial Biotechnology at ITEE-PIB was used. The studies were initiated by inoculating bacterial culture in LB medium (liquid broth). After 18 h, the culture was diluted in a ratio of 10^{-6}, in KH_2PO_4 buffer to obtain countable and visible colonies of *Bacillus subtilis* (ATCC, 6633). The membranes before the test were sterilized in a laminar cabinet using UV radiation for 30 min. Then, a portion of 10 mL suspension of bacteria was filtered at a pressure of 500 mbar in a *dead-end* set-up through unmodified (control) and modified membranes. The filtration membranes

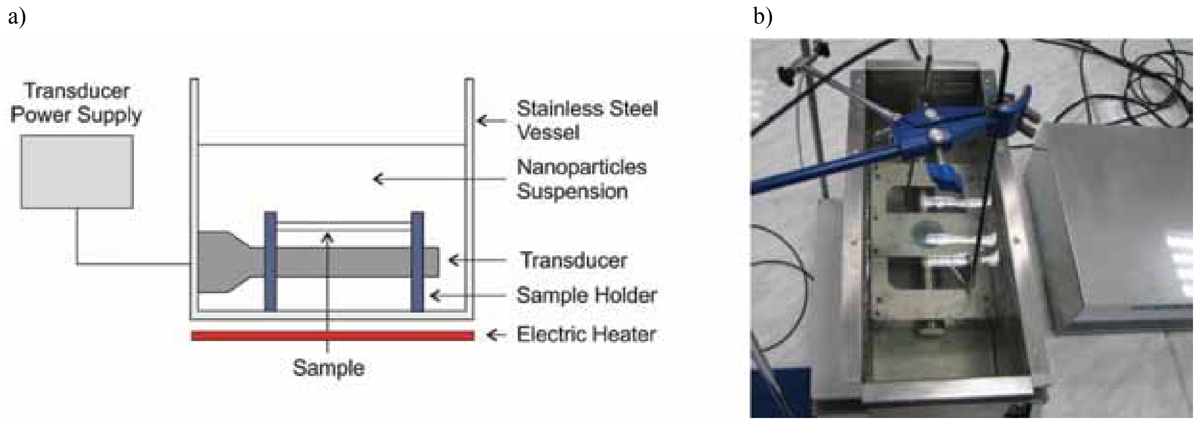


Fig. 2. Stand with tube transducer (a) and interior of the reaction chamber with the transducer and sample holder (b)

were placed on LB agar plate and incubated at 37°C for 24 h. After that, bacterial colonies that had grown on the membranes were counted. The results were expressed as a percentage reduction in the viability of the bacterial cells.

1.2.6. Leaching of nanoparticles

The modified polymeric materials were shaken at 150 rpm in 20 mL of demineralized water. The amount of zinc leached out from materials in demineralized water was measured every 30 minutes using an UV/VIS DR6000 spectrophotometer (HACH Lange) and cuvette tests (Hach, LCK, assay range: 0.2–6.0 mg/L) over a period of 6 hours and 30 minutes. The samples were then allowed to stand for 12 hours. Then they were shaken again in demineralized water for 3 hours, measuring the zinc concentration every hour. In the next part of the work, the samples were again left in demineralized water for 12 hours and shaken, measuring the zinc content every 2 hours until the minimal concentration was reached.

3. Results and discussion

3.1. Intensity of ultrasounds and cavitation

In the first part of the experiments, the ultrasound frequency was determined and their level and distribution in the reaction tank were examined. As a result, it was found that, for the PP stand, the ultrasonic frequency was 30.3 kHz and the level of ultrasounds and cavitation had a uniform distribution when measurements were made between transducers. These levels change, particularly in relation to cavitation when one or both transducers were operating (Fig. 3a). The OPCAV meter measured the levels in relative units in relation to the level of the reference signal recorded during meter calibration. Hence, the assessment of only changes in intensity and not its absolute value was possible. The measurement results indicate that the simultaneous operation of both transducers and the related acoustic signals interference does not cause disturbances of the cavitation level, which could result in a reduction in the efficiency of the nanoparticles deposition.

For the PT stand, whose frequency was 31 kHz, the measurements were made while moving the meter's probe along the transducer adjusting the speed of movement to the meter's time constant (signal sampling every 2s).

The measurement results indicate an increased level of ultrasounds near both edges and the centre of the transducer. This distribution was also confirmed by classical imaging of the intensity of the aluminium foil [20]. The transducer used in the study had a different construction compared to typical transducers used for welding, cutting, and disintegration [21], where the intensity was focused on the end of sonotrode.

3.2. The effect of time and temperature of the process

Nanoparticles of TiO_2 coated on the surface of filtration materials using the PP and PT stands had spherical structures (Fig. 4a and Fig. 4b), which aggregated into larger agglomerates increasing as the process time increased. This phenomenon was similarly observed for filtration materials modified with ZnO nanoparticle suspension.

The set results obtained by EDS for the same areas of the surface allow determining the tendency of layer growth, which is exponential for process times up to about 300 s (Fig. 5a). After this time, the damages of the membrane were found and the agglomerates increased along all axes.

A typical example of a layer formed under such conditions is shown in Fig. 4c. The uniform ZnO layer was interrupted only at the membrane failure sites. The damage mechanism was described by Masselin et al. [23]. The damages are mainly caused by compression

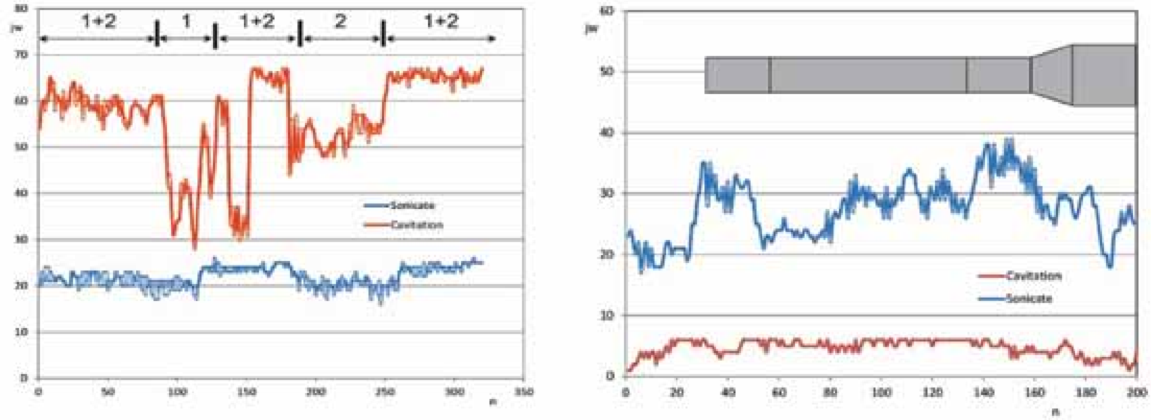


Fig. 3. The results of measurements of ultrasound and cavitation intensities, – relative units, n – measurement number: a) PP stand, 1 – T1 transducer on, 2 – T2 transducer on; b) PT stand – measurement along the transducer

and expansion of the material resulted from the passage of acoustic energy through the solid state materials and erosion caused by high velocity micro-jets, emitted during the implosion of cavitation bubbles at the surface of the sample. Damage leads to the loosening of filter material fibres, an increase in pore diameters, and the formation of large cracks affecting permeability and porosity.

Although the temperature increase affects the surface tension, viscosity, and saturated vapour pressure (parameters that influence the cavitation intensity), it did not have a clear impact on the intensity of the deposition of nanoparticles in the given temperature range (Fig. 5b).

3.3. Antibacterial properties

Samples of polymeric materials for testing their antibacterial properties were prepared using both stands, and the implementation of nanoparticles on the surfaces of filtration materials was carried out with the same process parameters (Fig. 6). The use of PP and PT stands

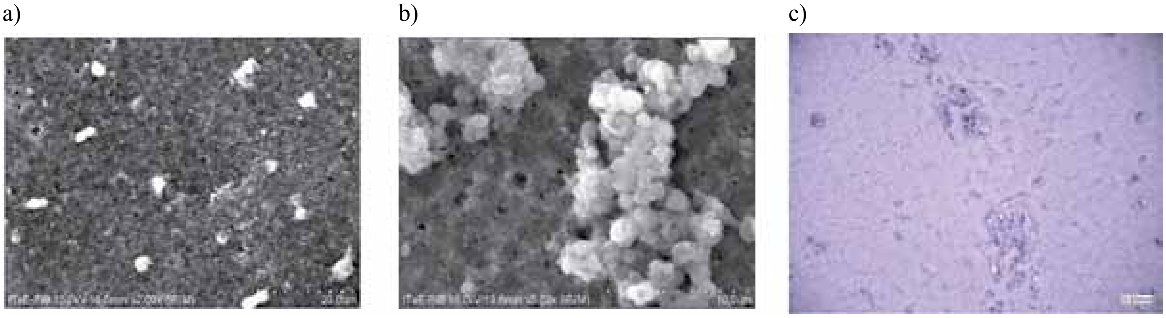


Fig. 4. The nanoparticles distribution on the membranes at different process times: a) SEM, TiO_2, process time 10 s, PP stand; b) SEM, TiO_2, process time 15 s, PP stand; c) Optical microscope, ZnO, process time 1200 s, PT stand

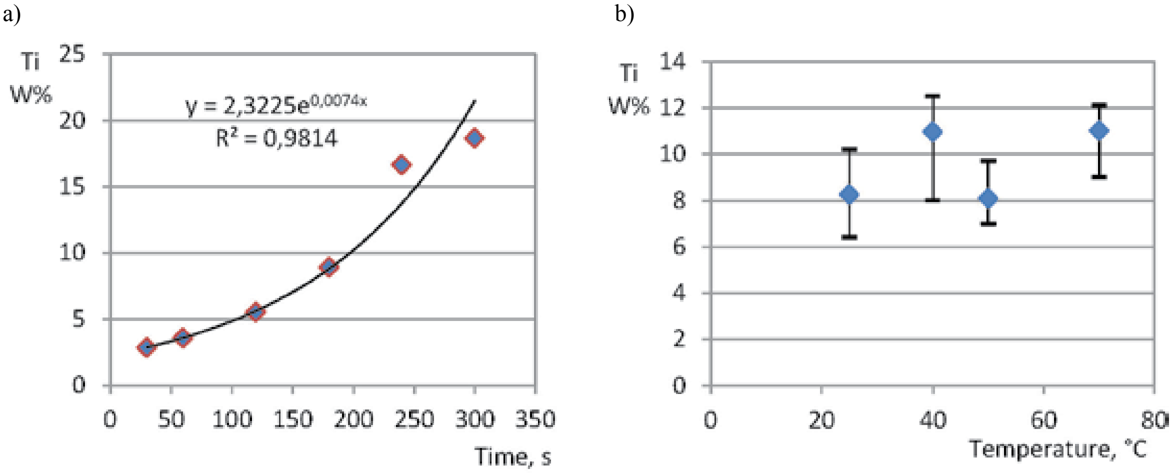


Fig. 5. Dependence of TiO_2 layer growth from time (a) and process temperature (b) average values with maximum and minimum value bars

Fig. 6. SEM imaging of samples selected for the tests of antibacterial properties: a) Filter, PP stand, ZnO, time 15s, Zn% Weight 11.17, temperature 40°C; b) Membrane, PT stand, ZnO, time 15s, Zn% Weight 3.62, temperature 40°C; c) Filter, PT stand, ZnO, time 15s, Zn% Weight 9.30, temperature 40°C

leads to a similar distribution of nanoparticles for both membrane and filter surfaces.

The results presented in Fig. 7 show the influence of ZnO nanoparticles on the viability of a representative strain of Gram-positive bacteria *Bacillus subtilis*, which due to cell structure is characterized by high resistance to biocidal agents and survival in a wide range of temperatures. Although the endospores contained in the cells (so-called spores) of *Bacillus subtilis* make them able to survive in extremely unfavourable conditions, membranes modified with ZnO nanoparticles using both ultrasounds generation stands produced a decrease in the amount of living bacteria by 99% on the material surface.

Studies on the antibacterial properties of modified filters (Fig. 6a and Fig. 6c) performed using *Escherichia coli* bacterial strain showed similar antibacterial properties. The percentage reduction in bacterial survival after contact with the modified material was 91% compared to the reference sample.

These results confirm that polymeric porous materials coated with ZnO via ultrasounds have antibacterial properties either for nano or micro sizes of compound structures deposited on materials [24]. The mechanism of ZnO interaction on bacterial cells is described by several phenomena, the main one of which is the generation of oxygen-reactive oxygen species on the oxide surface-hydroxyl radicals that cause damage to bacterial cells [8].

3.4. Resistance to leaching

The tests for the leaching of modifying agents from modified materials were conducted for samples from the same process in which ZnO nanoparticles were implemented on the filter surface at 40°C for 15 seconds at the PT stand. The process was subjected to the entire filter, and samples for the tests were taken from its surface. The lowest limit of the detection of concentration of Zn was reached after approximately 37 hours of the test.

EDS analysis of the same area of the samples before and after leaching showed that the amount of zinc decreased by 96.4%, and only 3.6% of the original amount remained on the surface, which was not enough to maintain the antibacterial properties of the filter performing an antibacterial test of the samples after the leaching tests were completed. Meantime, EDS analysis showed that agglomerates of ZnO with regular shapes and dimensions of 10 μm remained on the filter fibres (Fig. 8b and Fig. 8c). This indicates that the process has the potential to produce particles with good adhesion to the substrate.

Adhesive properties of the implemented nanoparticles are combined with spot melting of the substrate caused by the high speed and high temperature of nanoparticles ejected onto the solid surface by cavitation microwaves [8]. Improvement of adhesion can be obtained by carrying out the process at temperatures close to the melting point of the substrate or by supplementing the nanoparticle suspension with the component causing partial dissolution of the substrate. In both cases, however, there is a risk of damage to the membrane structure and degradation of its filtration properties.

Conclusions

The presented studies on the processes of ultrasonic implementation of nanoparticles on the surface of polymeric filters and membranes allow forming the following conclusions:

(1) Highly effective antibacterial properties obtained through the implementation of zinc oxide nanoparticles on the surface of polymeric materials can be used to control and prevent biofouling phenomenon of filtration membranes by destroying bacteria attaching to their surface.

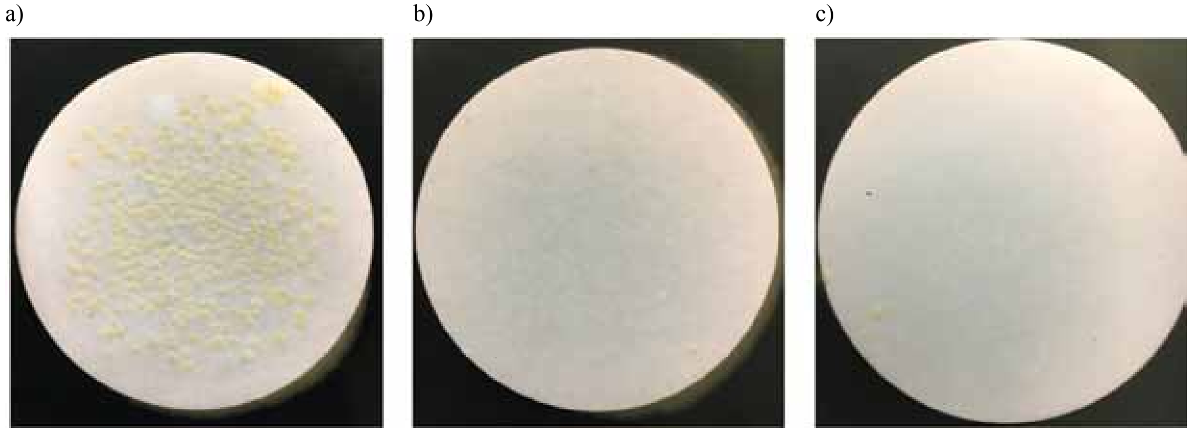
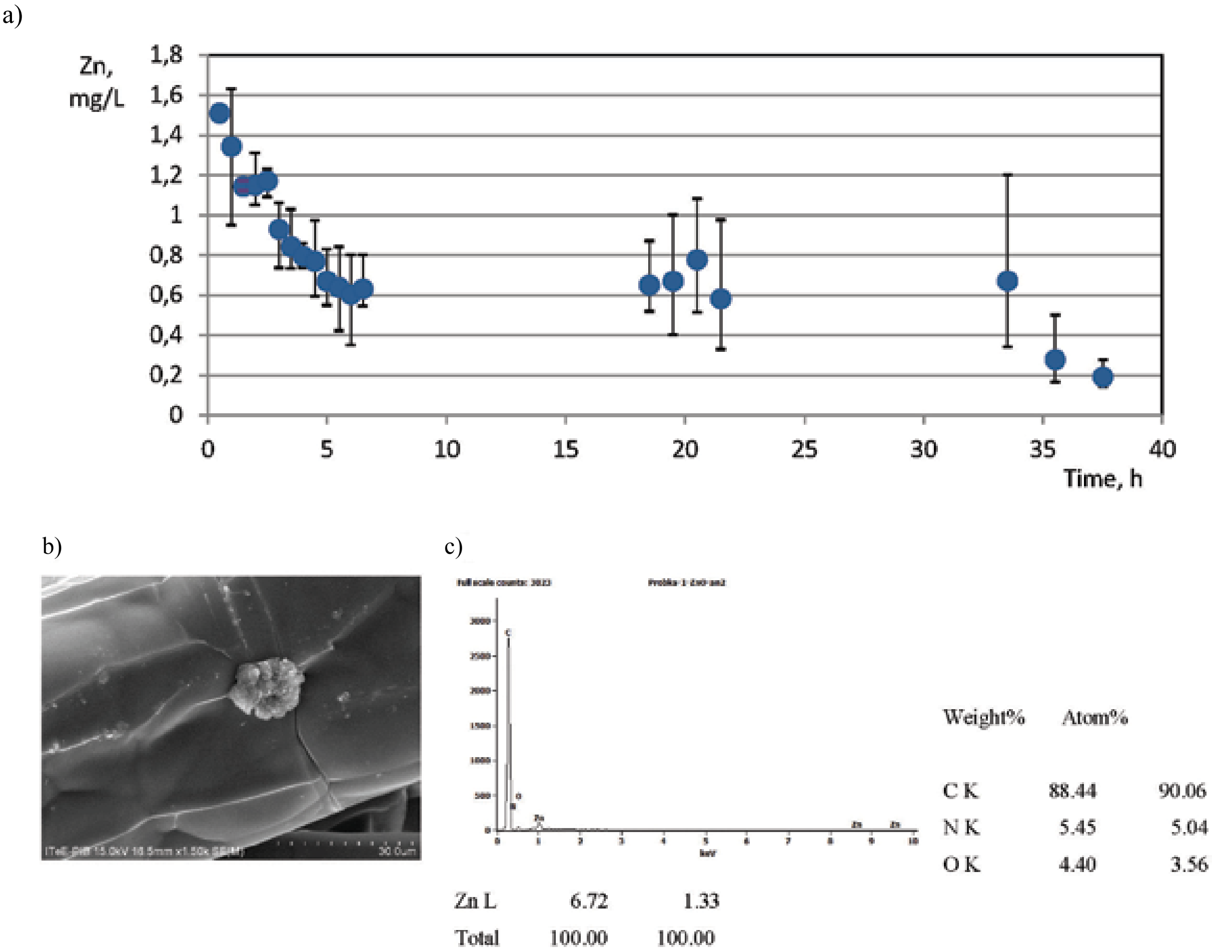


Fig. 7. Membranes after testing antibacterial properties: a) Unmodified membrane; b) Membrane after implementation of nanoparticles at the PT stand; c) Membrane after implementation of nanoparticles at the PP stand



	Weight%	Atom%
C K	88.44	90.06
N K	5.45	5.04
O K	4.40	3.56
Zn L	6.72	1.33
Total	100.00	100.00

Fig. 8. The results of the leaching resistance tests: a) Graph of average values of concentrations of eluted zinc; b) SEM image of a sample fibre with ZnO agglomerates after leaching test; (c) The results of the EDS analysis of the agglomerate

(2) Ultrasonic application of bioactive nanoparticles is a simple method, and it does not require complex apparatus. The one-stage application processes are short and energy-saving.

(3) The main factor determining the rate of deposition is the process time along with constant intensity of sound waves and the defined concentration of nanoparticles.

(4) The problem is the low resistance to leaching of deposited nanoparticles and the susceptibility of membranes to damages resulted from ultrasounds.

Further work should address the optimization of process parameters in order to improve resistance to the leaching of nanoparticles layers with a diminishing of the deterioration of filtration and separation performance of polymer porous materials, the evaluation of modified materials under real operating conditions, and analysis of the possibility of technology transfer to an industrial scale.

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