



Short communication

Removal of bacterial pathogen from wastewater using Al filter with Ag-containing nanocomposite film by *in situ* dispersion involving polyol processYoung Ik Seo^a, Ki Ho Hong^b, Se Hoon Kim^c, Duk Chang^d, Kyu Hwan Lee^e, Young Do Kim^{c,*}^a R&D Institute, Alutec Co., Chungcheongbuk-do 373-841, Republic of Korea^b Department of Advanced Technology Fusion, Konkuk University, Seoul 143-701, Republic of Korea^c Division of Materials Science and Engineering, Hanyang University, Seoul 133-791, Republic of Korea^d Department of Environmental Engineering, Konkuk University, Seoul 143-701, Republic of Korea^e Computational Science Center, Korea Institute of Science and Technology, Seoul 136-650, Republic of Korea

HIGHLIGHTS

- ▶ Ag/Al(OH)₃ mesoporous nanocomposite film was *in situ*-deposited on Al foam filter.
- ▶ The synthesized Ag NPs showed two distinct morphologies due to the Al(OH)₃ film.
- ▶ The Ag/Al(OH)₃ filter showed good bacteria removal rate within a short contact time.

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ABSTRACT

In this study, a filter with deposited Ag/Al(OH)₃ mesoporous nanocomposite film was fabricated to remove bacterial pathogens from wastewater. Mesoporous Al(OH)₃ film was generated on the Al foam body by alkali surface modification, followed by immersion in a polyol solution for 4 h at an elevated temperature in order to deposit silver nanoparticles (Ag NPs). The Al(OH)₃ porous matrix showed a significant increase in specific surface area due to the large size of the voids between flakes, which reached several tens of nanometers. After *in situ* three-dimensional deposition of Ag NPs by a polyol process, the Ag NPs were nucleated and grown at the surface of the mesoporous Al(OH)₃ film. The filter with Ag/Al(OH)₃ mesoporous nanocomposite film showed a good bacterial pathogen removal rate within a very short contact time compared to the untreated Al foam filter. Filters with deposited Ag/Al(OH)₃ mesoporous nanocomposite film have great potential for application as antimicrobial filters for tap water purification, wastewater treatment, and other bio-related applications.

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1. Introduction

Waterborne pathogens in drinking water including bacteria, protozoan oocysts and cysts, helminthes, fungi, rickettsiae, prions, and viruses have disastrous effects on public health, especially in developing countries of Africa and Southeast Asia; it has been estimated that 1.1 billion people do not have access to safe drinking water, 2.6 billion have little or no sanitation, and diarrhea kills over 6000 children a day from diseases transmitted through contaminated water or human excreta [1,2]. The U.S.EPA Science Advisory Board quoted drinking water contamination as one of the highest remaining environmental risks and microbial contamination as the greatest challenge in health risk management for

drinking water suppliers [3]. Therefore the value of water disinfection and microbial control are the most important tasks for both the health of human beings and the ecosystem. According to World Health Organization (WHO) recommendations, any water intended for drinking should contain zero fecal and total coliform counts in any 100 ml sample [4]. To adhere to the WHO recommendations for the wastewater treatment process, chemical agents (such as chlorine and its compounds) are most widely used because of their effectiveness and simplicity. However, this type of treatment takes a long time to get rid of bacterial pathogens, consumes a significant amount of chemical agents and there may be side-effects such as the formation of harmful disinfection byproducts, including carcinogenic trihalomethanes [5–7]. Nanotechnology may potentially overcome some of the aforementioned problems. For instance, Ag and Ag-containing compound nanomaterials, mostly in the form of nanoparticles and nanocomposites, have been intensively studied and developed for diverse bio-related applications such as long-lasting antibacterial protection when used in plastic products, and antimicrobial properties when used in medical dressing

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coatings for burn treatment [8–13] because of their strong cytotoxicity toward a wide range of microorganisms [14–18]. Silver has also been impregnated in filters in point-of-use tap water purification devices in order to retard the growth of microorganisms [19]. So, it can be deduced that the water purification filter which possesses Ag-containing nanomaterials can be more cost-effective way because of its semi-permanent characteristics than chemical agent method. The inhibitory effect of Ag^+ ions on bacterial growth is due to their adsorption to negatively charged bacterial cell walls, inactivation of enzymes, disruption of membrane permeability, and, ultimately, cell death [20–23]. Among various types of Ag-containing products, nanocomposites have been studied because diverse matrices can be used [12,13,24–29]. Nanocomposite matrices can be designed and manufactured to have an open porous structure for mass flow and direct contact between the biomass and the surface [17,18,20,21]. In addition, porous matrices are good substrates as they possess many nucleation and growth sites for fine dispersal of Ag NPs.

In this study, we deposited mesoporous Ag/Al(OH)₃ nanocomposite film on an Al foam filter using alkali surface modification and a polyol process to create a filter capable of rapidly removing bacterial pathogens from secondary effluent. Using alkali surface modification, we generated mesoporous Al(OH)₃ film and observed numerous pores with a remarkable increase in specific surface area due to the presence of flaky crystals woven within and covering the entire surface [30,31]. Using the polyol process as a three-dimensional deposition method, large amounts of Ag NPs were deposited on the surface of the porous matrix in direct contact with the external environment and therefore capable of reacting with bacterial pathogens. Al foam filters with deposited Ag/Al(OH)₃ mesoporous nanocomposite film can potentially be used as disinfection filters for tap water purification or wastewater treatment and as antimicrobial filters for biomedical applications.

2. Experimental details

The substrate, an Al foam plate (AA 6101, ERG Inc.), was cut into pieces with a diameter and thickness of 10 mm each. Sodium hydroxide (NaOH, 93%, Duksan Pure Chemicals Co. Ltd.) was used as a reagent for alkali surface modification. Pure water was obtained by double distillation followed by filtration with a Millipore Milli-Q plus purification system at an electrical resistivity of 18.2 MΩ cm. For three-dimensional Ag nanoparticle deposition, silver nitrate (AgNO_3 , 99.9%, Showa Chemicals Co. Ltd.) was employed as the Ag ion source, and ethylene glycol ($\text{C}_2\text{H}_6\text{O}_2$, 99%, Sigma-Aldrich) and polyvinylpyrrolidone (PVP, K-15, avg. MW of 10,000, Sigma-Aldrich) were used to reduce the Ag ions in the polyol process. All materials were used as received without further purification.

A simple alkali surface modification process was applied to the machined Al foam filter [30,31]. Alkali treatment was performed by immersing the filter in 5×10^{-3} M NaOH solution (pH 14) at 80 °C for 1 min. After the alkali treatment, the alkali-treated Al foam filter was immediately immersed in boiling water for 30 min to stabilize the surface-modified layer.

For three-dimensional deposition of the Ag NPs, the surface-modified Al foam filter was placed in a polyol solution and Ag NPs were deposited using the polyol process. The polyol solution was prepared using ethylene glycol solvent, PVP, and AgNO_3 precursor. First, PVP (1×10^{-4} M) and AgNO_3 (2×10^{-4} M) were added to ethylene glycol, mixed, and stirred at room temperature until they were completely dissolved. Then, the system was heated to 120 °C and held at this temperature for 4 h before rapid cooling in a water bath.

Table 1

The composition of *LES Endo Agar* used in this experiment (approximate formula per liter).

Component	Content (g)	Component	Content (g)
Yeast extract	1.2	Sodium chloride	3.7
Casitone	3.7	Sodium desoxycholate	0.1
Thiopeptone	3.7	Sodium lauryl sulfate	0.05
Pancreatic digest of casein	4.5	Sodium sulfite	1.6
Proteose peptone no. 3	3.0	Basic fuchsin	0.8
Lactose	9.4	Agar	15.0
Dipotassium phosphate	3.3	Monopotassium phosphate	1.0

The surface morphology of the filter was characterized by energy dispersive spectroscopy (EDS) using a field emission scanning electron microscope (FE-SEM, JEOL 6701F). To confirm the formation and crystallinity of Ag NPs in a mesoporous matrix, X-ray diffractometry (XRD, Rigaku RINT 2500/PC) was performed. To observe the cross-sectional characteristics of the nanocomposite film, transmission electron microscopy (TEM, TECNAI G2) was carried out at a high accelerating voltage of 200 kV. TEM samples were prepared for observation of cross-sectional morphology and selected area diffraction (SAD) patterns using a dual-beam focused ion beam (FIB, FEI Nova 600 Nanolab).

To characterize the antibacterial properties of the filters, a bath of secondary effluent (50 ml) containing *Escherichia coli* (*E. coli*) from a wastewater treatment plant was used. The secondary effluent was filtered using two types of Al foam filters: the original untreated filter and a filter which had undergone the previous procedure. *E. coli* were measured using a membrane filter technique as described in the APHA Standard Methods [32]. Subsequently, the membrane filter was placed on the *LES Endo Agar* (DIFCO Co.) which is the base for the enumeration of coliforms by the membrane filtration method as shown in Table 1, and incubated for 24 h at a temperature of 35 °C and a relative humidity of 60%. After incubation, the numbers of colonies on the membrane filter were counted by macrography.

3. Results and discussion

Fig. 1(a) shows photographs and scanning electron micrographs of the Al foam body at low magnification. The Al foam body possessed a porosity of around 93% and consisted of larger pores with a mean pore size of approximately 630 μm. Fig. 1(b) shows the smooth surface morphology of the untreated Al foam filter containing some residues. After surface modification, flakes covered the entire surface of the filter, as depicted in Fig. 1(c). The flakes had a thickness of around 20 nm and a width of several tens of nanometers. This surface is mesoporous and is characterized by voids between the flakes. In a previous study [30], we confirmed that for surface-modified specimens, the amount of mesopores in the size range of 10–100 nm was significantly increased and the specific surface area was increased by up to 10-fold compared to non-treated specimens. This mesoporous film can accommodate large amounts of Ag NPs because of the presence of numerous pores that act as nucleation and growth sites. After surface modification, the surface-modified Al foam body underwent a polyol process for 4 h at 120 °C. After the deposition of Ag NPs, numerous Ag NPs were present on the surface of the Al foam body, as illustrated in Fig. 1(d); in fact, the surface was entirely covered with a high density of Ag NPs. We attribute this finding to the attraction of Ag^+ ions to the hydroxyl groups (OH^-) of the mesoporous Al(OH)₃ film because of electrostatic attractive forces. These forces hold the Ag^+ ions on the surfaces of the pores in the matrix, and the ions can then be reduced to metallic Ag by a reducing agent such as ethylene glycol. The unique morphology of the matrix and its high specific surface

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