# PREPARATION AND CHARACTERIZATION OF FIBROUS NON-WOVEN TEXTILE DECORATED BY SILVER NANOPARTICLES FOR WATER FILTRATION

Petra PEER[1], Martina POLÁŠKOVÁ[2,3], Jaroslav MOSNÁČEK[2,4], Jana SEDLAŘIKOVÁ[3,5], Milan MASAŘ[3]

[1]*Institute of Hydrodynamics of the Czech Academy of Sciences, v. v. i., Prague, Czech Republic, EU, peer@ih.cas.cz*
[2]*Department of Polymer Engineering, Faculty of Technology, Tomas Bata University in Zlín, Zlín, Czech Republic, EU*
[3]*Centre of Polymer Systems, Tomas Bata University in Zlín, Zlín, Czech Republic, EU*
[4]*Slovak Academy of Sciences, Polymer Institute, Bratislava, Slovak Republic, EU*
[5]*Department of Fat, Surfactant and Cosmetic Technology, Faculty of Technology, Tomas Bata University in Zlín, Zlín, Czech Republic, EU*

**Abstract**

The environmentally friendly preparation of silver nanoparticles was proposed for the production of antibacterial non-woven textile used in water filtration. The silver nanoparticles were prepared by chemical reduction of silver nitrate using two environmentally friendly reducing agents, such as fructose or ascorbic acid. For comparison also commonly used reducing agent - sodium borohydride was used. The silver nanoparticles in various size and yield were produced by immersion of the plasma pre-treated polypropylene (PP) non-woven textile in the colloidal solutions for different periods (35, 45 and 135 min). The morphology of the silver nanoparticles was characterized by SEM and EDX analysis. Additionally, the antibacterial activity of the silver decorated PP non-woven textile was evaluated by an agar diffusion test using both Gram-positive *Staphylococcus Aureus* and Gram-negative *Escherichia coli*. The results suggest that the type of the reducing agent has major effect both on the morphology of silver nanoparticles and the antibacterial activity.

**Keywords:** Silver nanoparticles, reducing agent, non-woven textile, antibacterial activity

## 1. INTRODUCTION

Membranes for water filtration are capable of removing a wide variety of contaminants, ranging from large colloids, algae, bacteria up to individual ions, depending on pore dimensions [1-3]. Porous membranes accomplish separation essentially by size exclusion; solutes larger than pore size are rejected, while solutes smaller that membranes pore flow through pore structure. As the solutes build up on the surface or in the pores the productivity of membrane decreases, known as fouling. This process is often accompanied by accumulation and growth of microbial cells on surface and within the pores.

In principle there are two main approaches to the control of biofouling process. The first one is based on the modification of surface of the membranes by various methods, which makes the area less attractive for bacterial adhesion [4,5]. The second approach comprises membranes with the antibacterial agent, which either kills or inhibits the growth of microorganism and the quality and lifetime of the filter can be prolonged.

Among the large number of antibacterial agents available, silver nanoparticles attract the attention due to their low toxicity to human tissue and high activity against broad spectrum of bacteria, viruses and fungi [6,7]. In this context, several physical and chemical methods have been used for synthesizing and stabilizing silver nanoparticles (NPs), including chemical reduction using a variety of organic and inorganic reducing agents. Most of these methods are still in developmental stage and the stability and aggregation of NPs, control of crystal growth, morphology, size and size distribution represent the common experienced problems.

The aim of this study was production of silver/polyprolylene non-woven textile intended for the application as an antibacterial filter. For this purpose, the silver nanoparticles were prepared by chemical reduction of silver nitrate using two environmentally friendly reducing agents (fructose or ascorbic acid) and commonly used reducing agent (sodium borohydride). The effect of reducing agents and time of reduction was investigated in correlation with particle size and density of resulting silver nanoparticles. Besides, the antibacterial properties of non-woven textiles against the Gram-positive and Gram-negative bacteria were determined.

## 2. EXPERIMENTAL

### 2.1. Material and Preparation of Silver Nanoparticles

Silver nitrate ($AgNO_3$), fructose (F), ascorbic acid (AA) and sodium borohydride (SB) of analytical grade purity (Penta, Czech Republic) were utilized without further purification. *S. Aureus* (CCM 3953) and *E. Coli* (CCM 3954) were obtained from the Czech Collection of Microorganisms (CCM, Czech Republic). The bacterial cultures were grown on Mueller Hinton agar (HiMedia Laboratories, India).

In this experiment, 6.4 g of 1M $AgNO_3$ was dissolved in 750 mL of distilled water. Similarly, reducing agent, such as 0.5 g of fructose or 0.5g ascorbic acid or 19 mg of $Na(BH_4)$ was dissolved in 500 mL of distilled water. Then, the plasma pre-treated polypropylene (PP) non-woven textiles were immersed first in $AgNO_3$ solution and subsequently in solution of reducing agent for different time periods as described in **Table 1**. Before further analysis, the PP non-woven textiles were rinsed in distilled water three times to remove unfixed silver NPs. The experiments were proceeded at laboratory temperature.

**Table 1** Preparation of silver nanoparticles

| | | Immersion time in solution (min) | | |
|---|---|---|---|---|
| **Sample code** | **AgNO3** | Fructose | Ascorbic acid | $Na(BH_4)$ |
| F 35 | 5 | 30 | | |
| F 45 | 15 | 30 | | |
| F 135 | 15 | 120 | | |
| AA 35 | 5 | | 30 | |
| AA 45 | 15 | | 30 | |
| AA 135 | 15 | | 120 | |
| SB 35 | 5 | | | 30 |
| SB 45 | 15 | | | 30 |
| SB 135 | 15 | | | 120 |

### 2.2. Characterizations

Morphology of the silver nanoparticles sputtered by a thin gold layer was observed using a Vega 3 high resolution scanning electron microscope (Tescan, Czech Republic). The mean particle diameter and particle density were determined by help of Adobe Creative Suite software.

Elemental microanalysis was performed by the Octane SSD (area 30 $mm^2$) EDX (energy dispersive X-ray) detector (AMETEC, Inc) integrated into the NOVA NanoSEM 450 (FEI company) operated at 15 kV. The qualitative and quantitative results were obtained by applying the experimental procedure several times under

following conditions: distance between EDX detector and holder with sample - 5 mm, magnification of 10 000 or 30 000.

Antibacterial efficiency of silver NPs coated PP fibres against *S. aureus* and *E.* coli was tested by an agar diffusion method. Square (9 × 9 $mm^2$) samples were placed on agar plates pre-inoculated with 1 mL of 0.5 McF turbid bacterial suspension in sterile saline solution. The plates were then incubated at 37°C (24 hours) and after cultivation the diameters of inhibition zones around the samples were recorded in mm.

## 3. RESULTS AND DISCUSSION

Pre-treatment of polypropylene (PP) non-woven textiles was done in order to create the polar moieties on the PP fibres to anchor the silver NPs during the course of their preparation. This procedure should ensure the fixation of silver NPs on the surface of PP fibres and prevent their agglomeration at the same time. The plasma pre-treated PP non-woven textiles were immersed into the colloidal solutions for different periods of time (35, 45 and 135 min). The solutions turned light yellow immediately after immersion of PP non-woven textile to solutions of reducing agents, indicating the initial formation of silver nanoparticles. The SEM images of the PP non-woven textiles with silver NPs are shown in **Figure 1**. All reducing agents led to the formation of metallic silver ($Ag^0$) that was found on the surface of PP fibres and additionally confirmed by EDX analysis (**Figure 2**). A strong signal for elemental silver is noticed in the spectrum inferring the existence of metallic silver, in addition to the C peak originated from PP fibres.

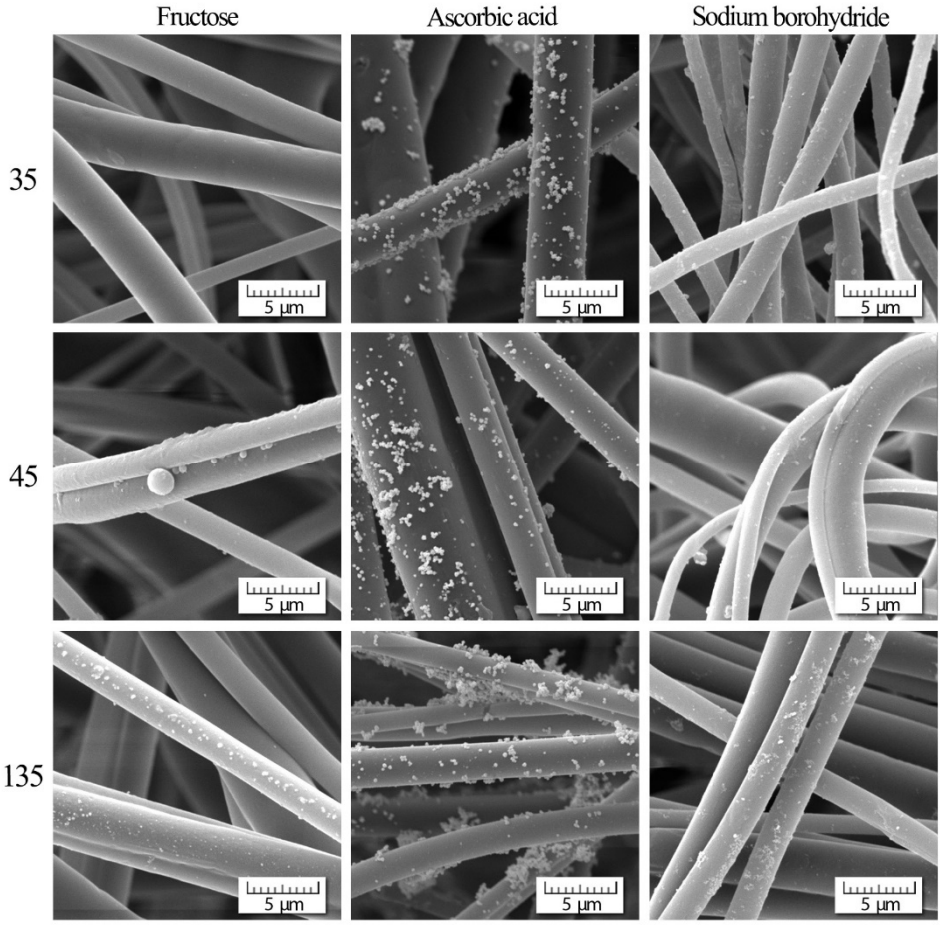


**Figure 1** SEM images of silver nanoparticles prepared using various reducing agents

It is visible that changes in time of immersion provided nanoparticles with various average sizes (**Table 2**). In the shortest immersion time, the silver NPs were spherical in shape, well separated and size ranged between

91 to 166 nm depending on the type of reducing agent. When the immersion time was prolonged, the silver NPs started to conjugate and aggregate into bigger particles up to 263 nm. At the same time, the density of silver NPs was increased with longer immersion time.

The reducing agent has significant effect on the size and density of the silver NPs on PP fibres. The most distinctive difference was found between two environmentally friendly reducing agents, fructose and ascorbic acid. The silver NPs from ascorbic acid exhibited the highest size and density, at the longest immersion time the silver NPs created veil of crystals between fibres.

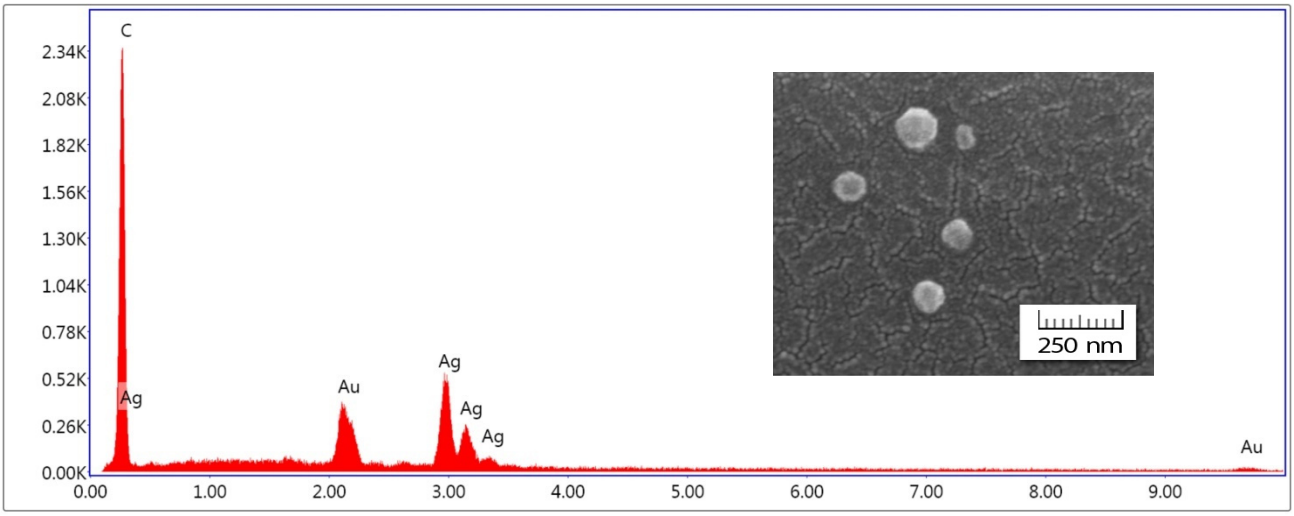


**Figure 2** EDX pattern of silver nanoparticles prepared by ascorbic acid as reducing agent (AA 35)

**Table 2** Size and density of silver NPs

| Sample code | Diameter of NPs[a] (nm) | Density of NPs[a] (counts/100 μm²) |
|---|---|---|
| F 35 | 91 ± 20 | 4.4 |
| F 45 | 138 ± 71 | 7.2 |
| F 135 | 169 ± 96 | 31 |
| AA 35 | 166 ± 55 | 62 |
| AA 45 | 189 ± 54 | 92 |
| AA 135 | 263 ± 75 | 101 |
| SB 35 | 118 ± 54 | 22 |
| SB 45 | 110 ± 37 | 45 |
| SB 135 | 137 ± 69 | 70 |

[a] area of 430 μm² was analysed

Results from antibacterial tests proved the activity against both representatives of Gram-negative and Gram-positive bacteria depending on the immersion time and type of reducing agent. The huge discrepancies in measured inhibition zones can be ascribed to the non-homogenous surface covering of PP fibres by the silver NPs. It can be also concluded that the shortest immersion time of 35 min was not optimal for creation of the sufficient amount of silver NPs to inhibit the growth of bacteria in all tested cases. The samples prepared using longer immersion times (45 and 135 min) exhibited clear zones ranging from 5.5 to 12 mm regardless the applied reducing agent.

## 4. CONCLUSION

The silver nanoparticles were prepared by different reducing agents on the plasma pre-treated PP non-woven textile. It was proved that the size and density of the formed silver NPs were influenced by time of reduction as well as by reducing agent, from which the ascorbic acid showed the most positive results. The density of silver NPs was high even at the shorter reducing period. Besides, the PP non-woven textile covered by silver NPs showed antibacterial efficiency against both representatives of Gram-negative and Gram-positive bacteria.

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