INNOVATIVE AND CLEANER INORGANIC TECHNOLOGIES
Module supervisor: dr hab. inz. Marcin Banach
Laboratory Instruction No 3
PROECOLOGICAL METHOD FOR OBTAINING SILVER NANOPARTICLES

REPORT:
ONE WEEK AFTER THE END OF THE CLASS

Author: Jolanta Pulit-Prociak

Projekt „Międzynarodowy program kształcenia Innowacyjne Technologie Chemiczne”
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Wydział Inżynierii i Technologii Chemicznej
Politechniki Krakowskiej im. Tadeusza Kościuszki
Ul. Warszawska 24
31-155 Kraków

tel/faks 12 628 20 35
tel. 12 628 27 01
www.chemia.pk.edu.pl

1. Introduction

Nanotechnology is concerned with the development and application of techniques that contribute to our understanding of various physicochemical phenomena. Its main task is to produce structures whose physical sizes are in the range of 1 to 100 nm (10 Å to 0.1 μm). Nanosilver (NAg) is one of the most useful commercial products belonging to the group of nanometals. Its application has recently reached the highest level of profitability. Like other nanoparticle structures, silver nanoparticles have been used in many broad areas of science. Antimicrobial activity is one of most widely known advantages of silver nanoparticles. This feature is mainly used in medicine, pharmacy, cosmetology and dentistry. There are many methods of obtaining silver nanoparticles. These include among others: chemical reduction, photochemical reduction, laser ablation, vacuum sputtering, microwave irradiation, electrochemical methods, and a number of biosynthesis-based methods.

1.1. Obtaining silver nanoparticles

Chemical reduction is one of leading methods for obtaining nanosilver. The high performance of the non-agglomerated nanoparticles, low price and ease of manufacture are its most valuable features. In addition, the process is generally carried out at atmospheric pressure, at room temperature or above but not exceeding 100°C.

The idea of this method is to perform a chemical reduction of silver ions with a reducing agent in the presence of a stabilizing substance. The purpose of this substance is to prevent the aggregation of silver nanoparticles. Particle size and their stability depends on many factors. These are: initial substrate concentrations, the molar ratio of the reducer and stabilizer to silver ions, the temperature and pH of the system. The stability of the suspension is determined by the electrokinetic potential (ζ). Under normal circumstances, in the

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gravitational field, nanoparticles do not aggregate because their sizes are so small that Brownian motion (chaotic heat) has more of an influence over them than gravitational motion. However, due to the collision of particles and their aggregation, Brownian motion gradually becomes weaker and the relative influence of gravity increases. This leads to the precipitation of solids. However, the diffusion layer that surrounds the colloidal particles, counteracts their aggregation. The diffusion layer is an ionic field that induces electrostatic repulsion between the dispersed particles. Electrokinetic potential, ζ is a way to measure this effect. The stronger the repulsion between the particles is, the higher the value of the electrokinetic potential. If the stabilizing agent belongs to the group of huge molecule substances, then steric stabilization is also possible.

2. Aim of experiment

The purpose of the experiment is to obtain four colloidal nanosilver suspensions and to collect their absorption spectra.

3. Chemical reagents and laboratory equipment

3.3. Laboratory reagents

- redistilled water
- silver nitrate
- reducing substance (see point 4)
- stabilizing substance (see point 4)

3.4. Laboratory equipment

- magnetic stirrer

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- water bath
- laboratory thermometer
- spectrophotometric cuvettes – set
- UV-VIS spectrophotometer
- glass reactor
- one-meter flasks (V = 250 ml)
- weighing cells
- beakers
- Pasteur pipettes
- one-meter pipettes
- 4 plastic containers

4. The course of the exercise

Table 1

Variant	Ag^+ source	System	Reducing agent			Stabilizing agent			Temperature [°C]
1	$AgNO_3$, 50 ml, 0.001 M	1	Ascorbic acid	0.002 M	25 ml	Natural protein stabilizer (gelatin), 25 ml, 0.8%			50
		2		0.006 M	25 ml				
		3		0.011 M	25 ml				
		4		0.02 M	25 ml				
2		1	Ascorbic acid 0.011 M, 25 ml			Natural protein stabilizer (gelatin)	0.004%	25 ml	50
		2					0.4%	25 ml	

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		3			0.8%	25 ml	
		4			2.4%	25 ml	
3		1	Ascorbic acid 0.011 M, 25 ml	Natural protein stabilizer (gelatin), 25 ml, 0.8%			25
		2					40
		3					50
		4					80

Prepare aqueous solutions of silver nitrate, stabilizing and reducing agents. Solutions of silver nitrate and reducing agent should be prepared in one-meter flasks and a solution of the stabilizing substance should be prepared in a beaker. In order to dissolve all of the total gelatin, the contents of the beaker should be stirred and heated.

Measure 50 ml of $AgNO_3$ solution into a glass beaker which should be put into a water bath placed on magnetic stirrer. Add 25 ml of the stabilizer solution and mix them until desired temperature is reached. Then, introduce 25 ml of reducing agent solution and continue stirring for another 5 minutes. After this time, disconnect the reactor vessel from the mixing and heating system and transfer the resulting suspension to a plastic container. Proceed along the same lines for all experiments, according to Table 1.

5. Characteristics

The obtained suspensions should be diluted prior to spectrophotometric analysis in order to avoid too-high-concentration effects. For this purpose, transfer 1 ml of prepared suspension to a plastic vessel and add 9 ml of redistilled water. Mix thoroughly. Then, perform a spectrophotometric analysis of the diluted suspensions. Fill the spectrophotometric cuvette with 3 ml of analysed suspension. In a similar way, prepare a reference cuvette by filling it with redistilled water. In the event of air bubbles appearing, they should be removed with a Pasteur pipette. After wiping the cuvette with a cloth, place it in the spectrophotometer and close the cover of the apparatus. Then, after setting the measuring holder in such a position that the light beam passes through the cuvette, a reference sample shall be measured. To do so, press the "START" button in the UVSoftware [Spectrum Scan] window. At the end of the reference measurement, the

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position of the cuvettes should be changed, allowing the light beam to pass through the tested cuvette. In the dialog box press "OK" and the measurement will start automatically. Save the spectrum by pressing "DATA" and "SAVE" (folder: Student labs * Team * *).

6. Report

The report should include: an introduction (from 0.5 to 1 pages with citations), course of work, results and their analysis (UV-VIS spectra should be interpreted according to calculated molar ratios of reducing substance to silver ions or amounts of stabilizer used or temperatures of experiments), a summary with conclusions and a list of references.

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