Gold nanoparticles evaluation using functional optical coherence tomography

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ABSTRACT

The main object of this research was to assess the ability to characterize the gold nanoparticles using optical modalities like optical coherence tomography. Since the nanoparticles, especially gold one, have been very attractive for medical diagnosis and treatment the amount of research activities have been growing rapidly. The nanoparticles designed for different applications like contrast agents or drugs delivery change the optical features of tissue in different way. Therefore, the expanded analysis of scattering optical signal may lead to obtain much more useful information about the tissues health and the treatment efficiency. The noninvasive measurements of the concentration and distribution of the nanoparticles, as well as their size in the media have been taken under consideration. For this purpose the polarization sensitive optical coherence tomography system with spectroscopic analysis (PS-SOCT) has been designed and used. In this contribution we are going to present the PS-SOCT measurement data obtained for the gold nanoparticles. The measurements have been taken for the liquid (gold nanoparticles in water) samples changing the particles concentrations in time.

Keywords: optical coherence tomography, polarization and spectroscopic analysis, gold nanoparticles

1. INTRODUCTION

Since the last decade the nanoparticles have been widely applied in science and industry, as well as in biomedical applications and medical treatments. For many reasons the gold nanoparticles have been used as the contrast agents for biomedical imaging [1][2]. Moreover, they can be applied successfully for the tumors detection and delineation of cancer affected area [3][4]. However, for biomedical imaging the common approach is to use the gold nanoparticles mostly for imaging contrast enhancement. Despite of great achievements in nanotechnology the nanoparticles influence on optical properties of scattering media like tissues and their potential usefulness for medical treatment are still of word wide research interests. The aim of this work was to analyze the influence on the scattered optical signal of the gold nanoparticles. In this case different sizes, different concentrations and distributions of the nanoparticles in liquid samples have been measured. Moreover, the agglomeration process of the particles has been monitored for liquid samples, as well as their surface plasmon resonance changes. All the collected data includes full characterization of the backscattered light from particular points inside the evaluated samples consists of spectroscopic and polarization sensitive analysis. For this purpose a homemade polarization sensitive optical coherence tomography system with spectroscopic analysis (PS-SOCT) has been used. Beside the intensity OCT images this system is capable to deliver the map of local optical anisotropy changes in the form of retardation angle, as well as backscattered light spectral characteristic for each scattering center. The combination of both polarization sensitive and spectroscopic modalities are also possible. The PS-SOCT has been described in details, as well as signal processing methods, in [5-8]. Such set of measurements should be useful to find a correlation between OCT data and gold nanoparticles size, form and volume distribution.

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2. THE IDEA AND PROOF OF CONCEPT

The experiment has been carried out in order to express the changes of spectral characteristics and polarization state of light backscattered from nanoparticles obtained from optical coherence tomography (OCT) measurement data. As a device under tests the glass capillaries filled with gold nanoparticles dispersed in water were used. The water suspension of gold nanoparticles was prepared in the laser ablation in liquid process described in our previous work [9]. The size in diameter of the nanoparticles, determined by means of laser diffractometer as well as calculated on the basis of spectroscopic data, were below 50 nm. All measurements has been made by the use of the polarization sensitive spectroscopic optical coherence system (PS-SOCT) designed and developed at the department of Metrology and Optoelectronics at Gdańsk University of Technology. For the experiment three samples of water suspension of gold nanoparticles have been prepared. Two of them (sample 1 and 2) have been modified by means of laser post irradiation process in order to modify average nanoparticles diameter as well as inducing agglomeration process, resulting in shifting resonance absorption band. Increasing nanoparticle diameter shifts the Surface Plasmon Resonance (SPR) into longer wavelength what is presented in Fig. 1. Unmodified sample (3) was used as a reference.

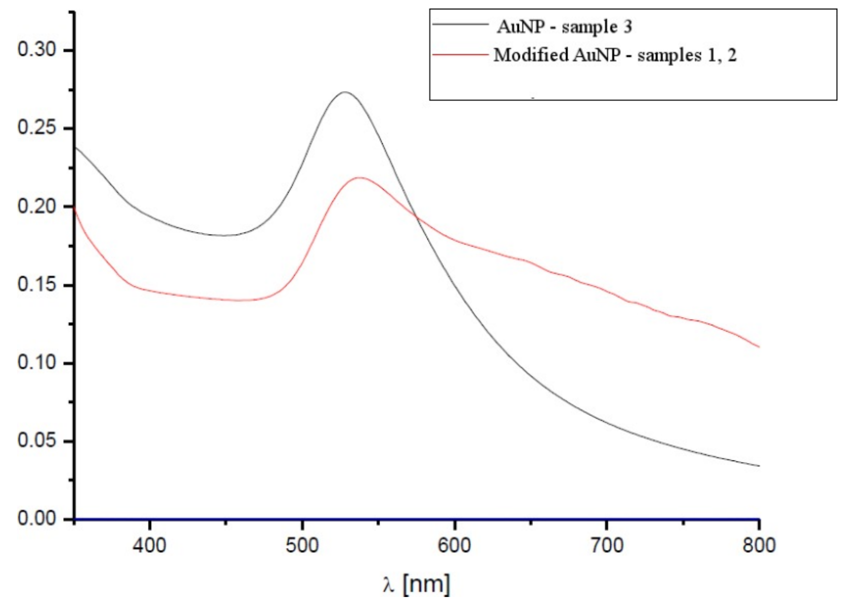


Fig. 1. Absorption spectra of gold nanoparticles suspension in deionized water prepared for the experiment, The absorption spectra of samples 1 and 2 were similar at the beginning after samples preparation. However, they have changed their features differently in time, which has been expressed based on experimental data

For samples (1) and (2) the scattering coefficient in the long wavelength range has been modified and was high enough to produce backscattered optical signal in NIR range, which could to be recorded by OCT technique. However these samples had a very low stability in time, which caused the nanoparticles trend to agglomerate. This effect has been also monitored in time. The reference sample (3) characterized with maximum of SPR in the visible range (530 nm) was the stable one having a very low scattering features in IR range. To predict the samples behavior and OCT measurement results the numerical analysis of the scattering phenomena of gold nanoparticles using Mie theory has been made. Theoretical analysis was performed for two nanoparticle diameters 50 nm and 100 nm dispersed in deionized water. We calculated optical properties for several particles volume concentrations: 0.05% V/V, 0.1% V/V, 0.2% V/V, 0.3% V/V, 0.4% V/V, 0.5% V/V. Based on obtained data the backscattering light extinction coefficients were estimated and presented in Fig 2.

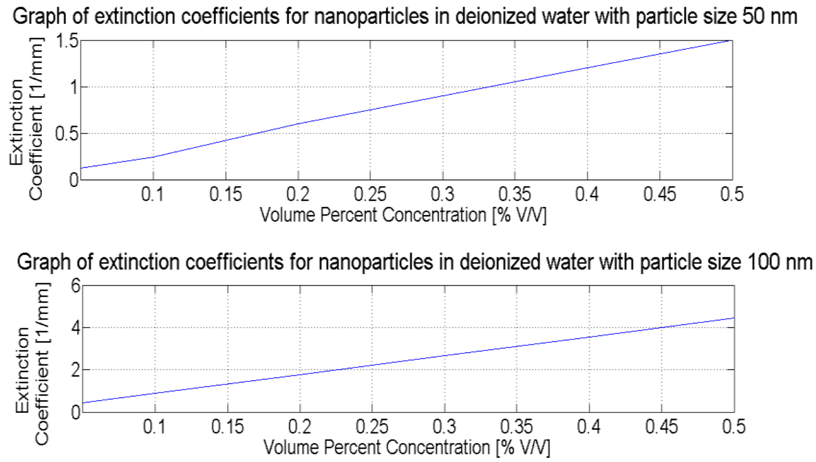


Fig. 2. Graphs of extinction coefficients for two types of gold nanoparticles in deionized water

Presented results of calculations (Fig. 2) show a strong relation between scattering and absorption features, particles size and volume concentration. For small particles under the 50 nm (sample 3) the backscattered signal would be too low to be recognized. However for agglomerates of nanoparticles (samples 1 and 2 can be detected successfully using optical coherence tomography techniques. This assumptions was confirmed based on OCT measurements data.

In our research, aside from obtained images of materials internal structure and quantitative analysis of optical coefficients, polarization sensitive and spectroscopic analysis have been done. For the experiment the standard, polarization sensitive OCT system has been used. This system has been equipped with the data acquisition and analysis software, developed in our laboratory (at Department of Metrology and Optoelectronics, Gdansk University of Technology) [6-7]. The additional features of the software deliver the information about the intensity of the light backscattered for particular points inside the tested object, its state of polarization, as well as spectral characteristic for spectroscopic analysis. Its measurements features have been summarized in Table 1.

Table 1. PS-OCT system features.

Item	**Value**
Light source type	20 kHz swept source (SS)
Average output power	10 mW
Central wavelength	1320 nm
Wavelength range	140 nm
Axial resolution	12 μm
Lateral resolution	15 μm
Frame rate	> 4 fps
Max. depth imaging range/ transvers imaging range	7 mm / 10 mm

The results of polarization sensitive OCT measurements of these three samples taken at the beginning and after two days of the experiment have been presented in the Fig. 3. The measurements were made by the use of glass capillaries.

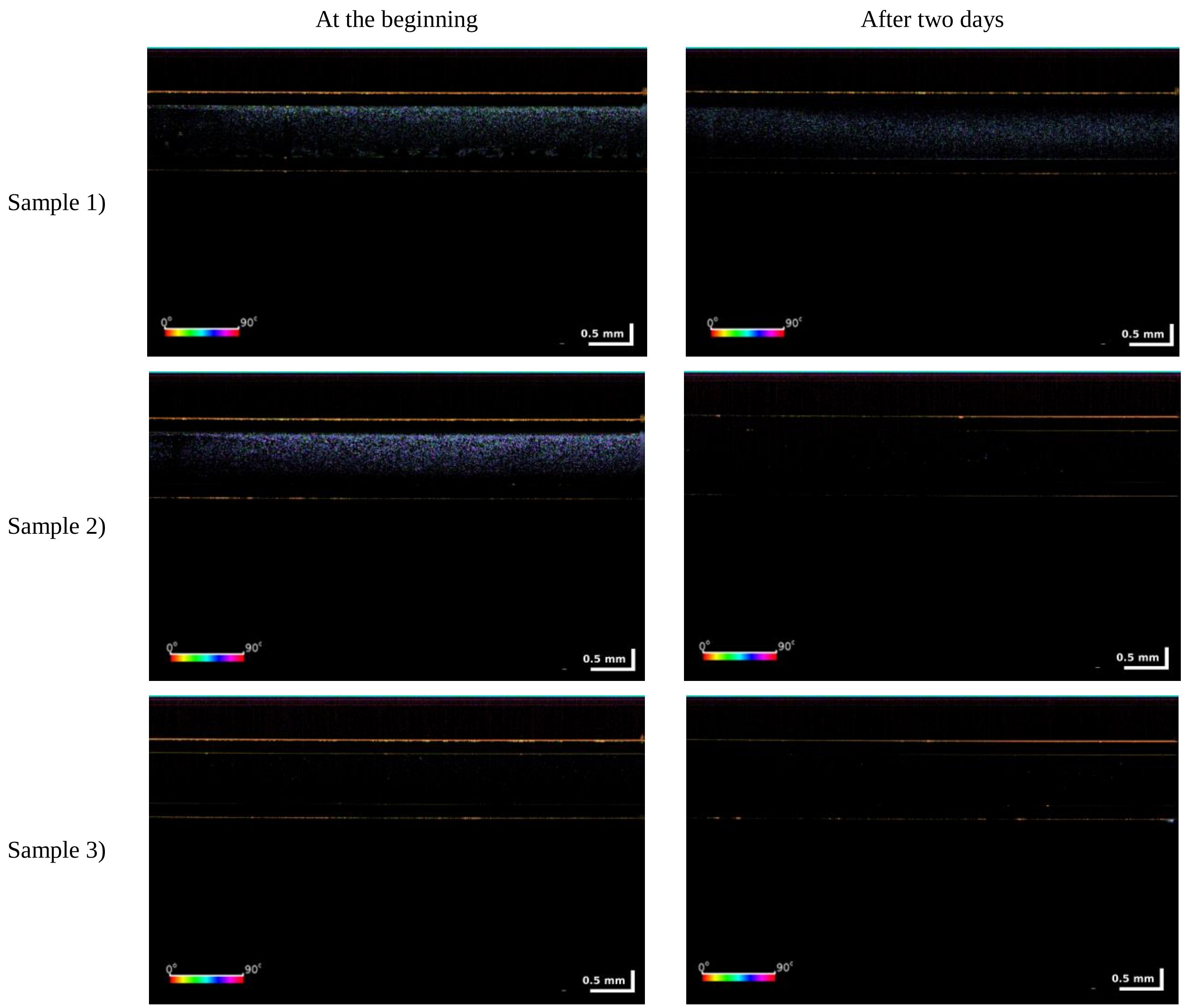
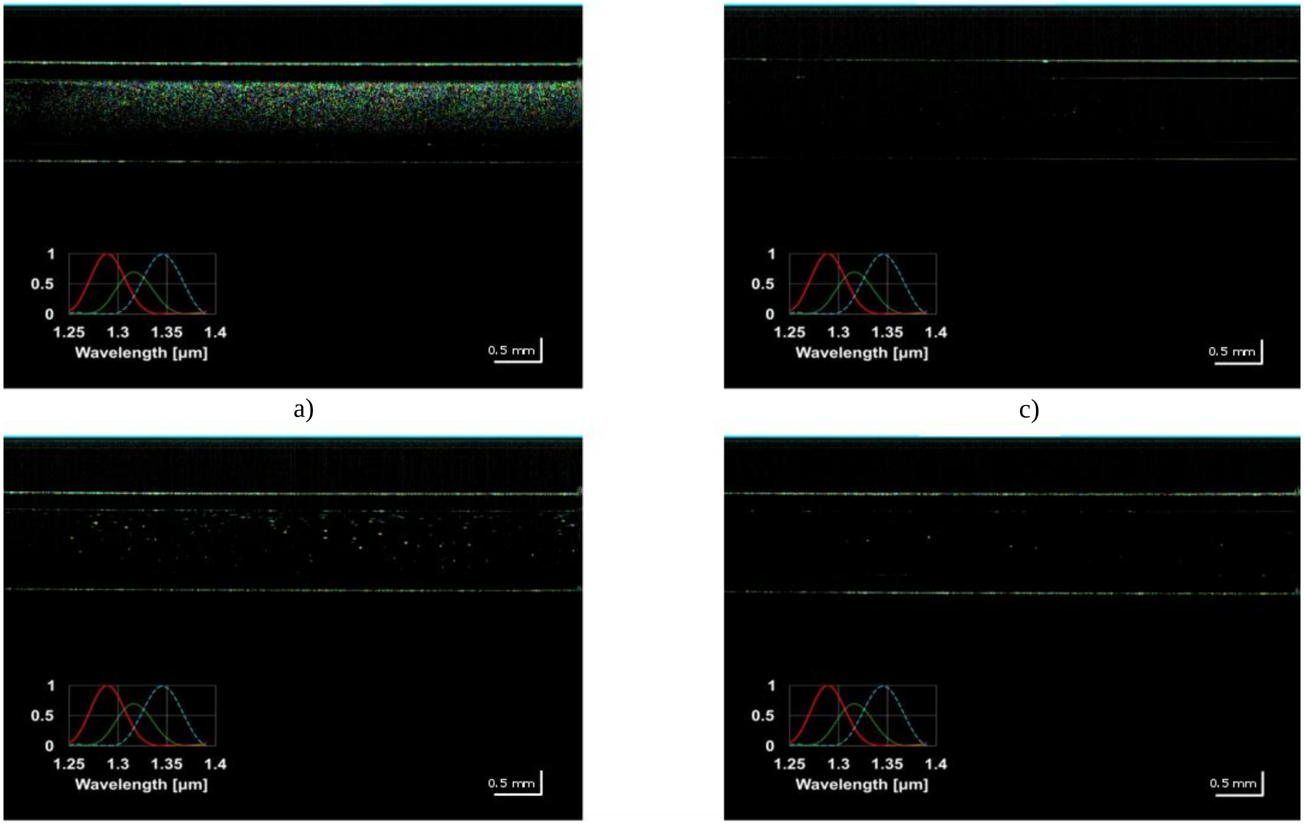


Fig. 3. Retardation OCT images of water suspensions of gold nanoparticles.

Presented OCT measurements results of the samples have shown the changes of scattering features in time. The reference sample (sample no. 3) has not given any difference in intensity and polarization OCT images. However, analyzing images taken for samples 1 and 2 one can observe quite high backscattering effect, which drops in time. The higher scattering can be explained by the presence of gold particles agglomerates. After that some heavier complex structures just felt down to the bottom of the sample, which reduced the overall number of gold particles in the suspension volume. This can be seen especially for sample 2, where after two day of the experiment the OCT images looked almost the same as for the reference one (sample 3).

Similar measurements were made for the sample 2 using spectroscopic analysis in OCT. The spectroscopic analysis extracts spectral information about backscattered light from the OCT measurements. This method increase the imaging contrast, as well as can be useful for particles size estimation. The spectral information has been codded in OCT images using different colors of pixels, which correspond to backscattered light spectra. Obtained results have been presented in Fig 4.



a) c) b) d)

Fig. 4. Spectroscopic OCT images of water suspensions of gold nanoparticles (sample 2) recorded in the following days; a-d images from each day starting from the first one (a).

The trend of changes has confirmed the conclusions, which have been made for results presented in Fig. 3. At the beginning the backscattered signal has been recorded from every point inside the sample. The colors of the pictures cover the range from red to blue, however the most of them were placed in green and blue region. Based on scattering theory the highest scattering effect for short wavelengths is observed for smaller particles. Therefore, the spectral characteristic of backscattered light also depends on particles size [10-11]. If the particles have a strong ability to agglomerate the population of the smallest one is being reduced (red color of pixels). Comparing images taken at the beginning and after one day (Fig. 4 a and b) one can see that in the image marked as b the pixels are reddish or greenish with just a few of blue one. It means that only small particles remains in the solution, while the rest were removed by falling down to the bottom of the sample.

Based on OCT measurement data the average value of extinction coefficients of each sample as a function of time were calculated. It is important to note, that extinction coefficient have been specified for suspension, not for single nanoparticles. Obtained results were presented below (Fig. 5)

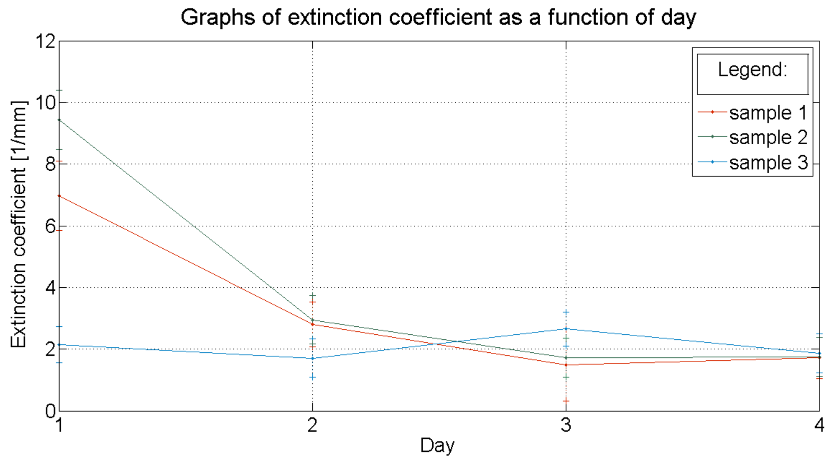


Fig. 5. Graphs of mean value of extinction coefficient as a function of day for three samples with gold nanoparticles suspension

The characteristics in Fig. 5 present mean value with standard deviation of extinction coefficient of each sample. Apart from volume concentration (the same at the beginning in every samples), a low value of extinction coefficient for sample 3 are in good agreement with numerical calculations and experimental observations. While the bigger agglomerates appear the scattering level drastically increase. Moreover, the drop of extinction value in the following days are caused by drop in number of particles in sample volume.

3. CONCLUSIONS

Functional optical coherence tomography opens new spaces for optical measurements of materials and structures composed with nanodopants. Here the usefulness of OCT enhanced with polarization sensitive and spectroscopic analysis have proven for evaluation of materials with gold nanoparticles. Compering to other measurement techniques like SEM (Scanning Electron beam Microscopy) or AFM (Atomic Force Microscopy) OCT will newer gives such detailed images with nanometer resolution. However, using functional extensions and statistical analysis the size and concentration of nanoparticles can be estimated. Moreover, the whole sample can be evaluated efficiently, where the SEM and AFM are limited only to measure the sample surface. Besides, the OCT method is fast, safe for the tested device and does not need a special conditions like sample preparation to record high quality tomographic images.

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