Synthesis and analysis of Nanoparticles with the low-cost measuring system LabPi

Björn Bartram, Timm Wilke, Manuel Wejner

Friedrich-Schiller-Universität Jena, Institute for Inorganic and Analytical Chemistry, Chemistry Education Department, Germany

Abstract

As nanotechnology and nanomaterials continue to be further developed, particularly in nanomedicine, not only are new approaches being researched to treat complex diseases, but they are also becoming available in large quantities for everyday use. This accessibility offers many novel possibilities to contextualize nanomaterials in applications for hygiene and medical products, such as cleaners, dressings, and plasters. Weighing the potential versus the risks of using this technology in everyday products provides multiple learning opportunities, which have already been presented in some articles about silver nanomaterials [1, 2]. The focus in chemistry classes here is often on synthesis, unique properties, the possible benefits, and the potential hazards to humans and the environment.

Further, more in-depth learning opportunities in chemistry lessons can be initiated with the integration of modern instrumental analysis methods. On the one hand, the use of low-cost technology can make otherwise cost-intensive equipment such as spectrometers more accessible in STEM lessons, and on the other hand, the digital networking of this equipment can be used to discuss the measurement results and their relevance in a collaborative approach. The LabPi measuring station serves as an example of such a cost-effective solution [3].

The combination of nanotechnology and laboratory measuring stations thus enables better connection of current research topics with related analytical methods such as like spectro- and photometry. In turn, this creates further learning opportunities for meaningfully embedding the topic of nanotechnology in chemistry lessons and thereby deepening knowledge of important chemical analysis methods. In this article, the quantitative determination of silver nanoparticles using commercially available silver nanoparticles will be compared to those from a synthesis suitable for school use and presented for photometric determination in an everyday product.

Keywords: *Nanotechnology, Measurement Systems, LabPi, Digitalization, STEM Education 4.0*

1. Introduction

The continuous development of new (nano-)materials represents a great opportunity to overcome current and future problems of our living environment. From rapid corona tests based on gold nanoparticles, to new approaches with silver nanoparticles to combat multi-resistant germs or modern cleaners – this technology is increasingly being applied in a variety of useful, but also questionable contexts. The reflection of this application is essential to prevent damage to humans and the environment and is therefore a suitable topic for STEM lessons to promote the evaluation competence on current examples. For this purpose, nanomaterials and their properties can be used for many other contexts, such as quantitative determination via photometry.

Due to the availability of various low-cost sensors and the good networkability of devices, technical possibilities are also becoming increasingly accessible, so that more complex metrological infrastructures can be set up or created in schools. In this way, measurement methods such as spectrometry, polarimetry or gas chromatography can be implemented for schools even on a small budget [3].

2. Objectives

There are already scientific contributions on applications and properties of silver nanomaterials in chemistry education, addressing different contexts like the antimicrobial effect of silver nanoparticles [1,2]. Spectro- and photometry will now be related to this context. On the one hand, the analytical procedure is to be presented on a current research topic and its basic features are to be conveyed. On the other hand, a conception of the effective quantity dimension of nanomaterials is to be developed. To demonstrate the reference, this will not only be carried out using self-produced silver nanoparticles, but also with a freely available cleanser.

3. Didactical Reconstruction

In scientific research, nanomaterials are synthesized in a bottom-up approach, purified using lengthy separation procedures, and then characterized using various methods. For example, UV-Vis spectra, dynamic light scattering or scanning electron microscopy are used to determine the amount and size distribution of nanoparticles by several methods. Since nanoparticles, especially in dispersions, are subject to various aging processes that change their properties over time, they also have a limited shelf life and the data collected are only reliable for a certain period.

When teaching nanomaterials, such methods for purification and investigation are difficult to implement in terms of time and, because of the equipment, can often only be carried out in cooperation with a university. However, a synthesis or acquisition and thus a thematization in chemistry classes is not impossible. The fact that nanoparticles can be synthesized in the classroom or student laboratory and are suitable for model experiments to illustrate potential applications or hazards has been sufficiently documented in the literature.

Due to a wide variety of (digital) approaches, a wide range of analytical methods are also possible for investigation, which in their further development will make even more options available for chemistry teaching in the future. Notable examples of this are LD Cassy [4] or the low-cost measuring station LabPi [5], which are being developed with didactic objectives and for use in STEM teaching. These lowers the (important) financial barrier and allows access to important sensor technology and measurement devices, such as spectro- or polarimeters [3,5].

In the following, the chemical preparation of silver nanoparticles is presented first. Subsequently, these nanoparticles are investigated spectrometrically using the LabPi measuring station. To control the quality of the nanoparticles and the measuring device, commercially purchased particles and a research-grade UV/Vis spectrometer are contrasted. Finally, synthesis and investigation will be piloted with students (**Fig. 1**).

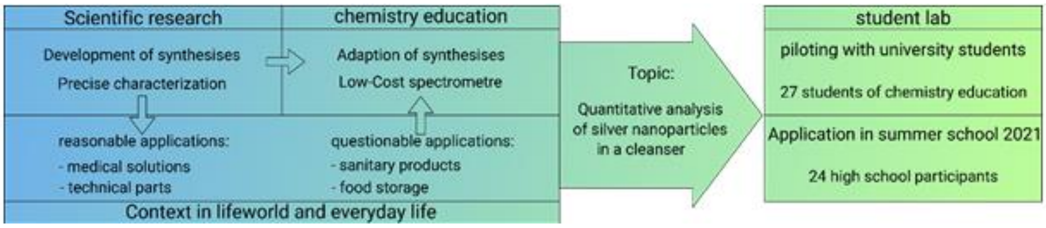

Fig. 1: Didactical Reconstruction and research design.

3.1 Synthesis of silver nanoparticles

A well-established approach an silver nanoparticle synthesis according to Kittler et al [6] or Wang et al [7] is carried out with the aid of a silver nitrate solution (0.1 mol/L), which is reacted with the aid of 2 g D(+)-glucose and 1 g polyvinylpyrrolidone. After 60 min under heating to about 90 °C, the yield according to literature is 1 m% of silver nanoparticles based on the amount of silver nitrate used, with a size of about 50 ± 20 nm [6]. The formation of the nanoparticles can also be observed with the naked eye: The initially colorless solution turns increasingly intense yellow/orange over time. According to literature, the dispersion prepared in this way has a theoretical concentration of 22.88 mg/L. For comparison, silver nanoparticles (Econix Silver Nanospheres - PVP (Dried) of nanocomposix 50 nm, 40 kDa PvP) were purchased and obtained as a dark solid. A stock dispersion was prepared with these at a mass concentration of 25 mg/L. To ensure that the nanoparticles are well dispersed, the dispersion is placed in an ultrasonic bath for 60 seconds.

3.2 Spectrometric characterization

To analyze the silver nanoparticles, UV/Vis spectra are recorded. The reason for the colorfulness of noble metal nanoparticles is the so-called plasmon resonance. In this process, the charge of a nanoparticle oscillates at a certain frequency (the so-called plasmon), so that irradiated light of a specific wavelength is extinguished. This extinction depends on the concentration, but also on the size and shape of the nanoparticles, so that not only the quantity can be approximated, but also limited statements about the size and shape of the nanoparticles can be made. For spherical silver nanoparticles, the extinction wavelength is between 395 and 520 nm [8]. Reactants and by-products still present in the prepared dispersion are not active in visible light, so their influence on the measurement can be excluded [7]. Using the purchased silver nanoparticles, which have been purified by the manufacturer and have a measured size distribution, it is now possible to spectrometrically check whether the size and yield of the synthesized nanoparticles correspond to the literature values (**Fig. 2**).

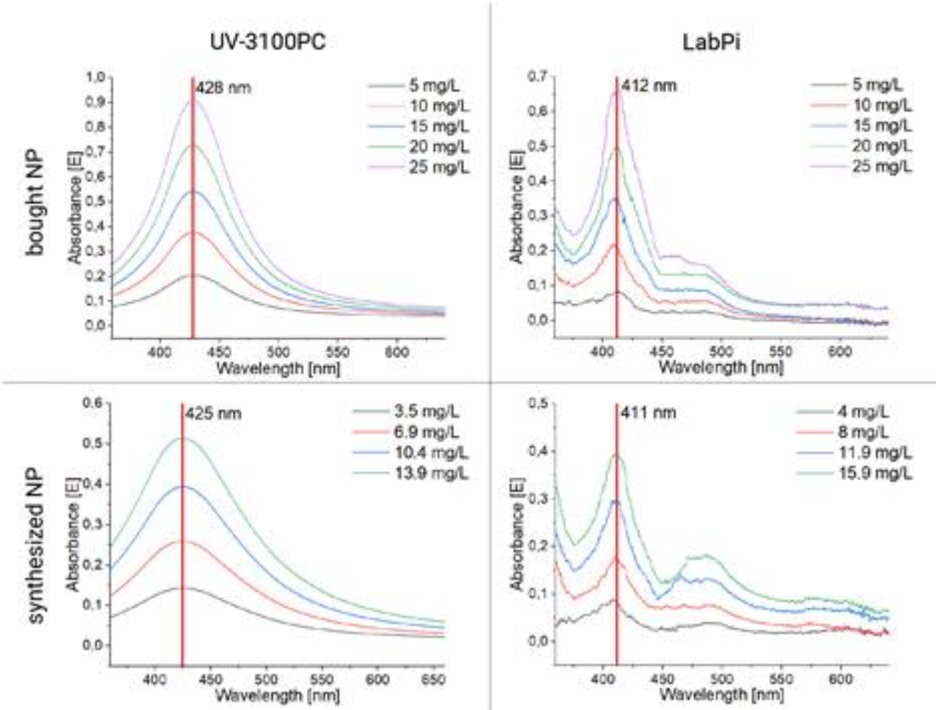

Fig. 2: UV-Vis spectra for silver nanoparticle dispersions at different concentrations (VWR Spectrophotometer UV-3100PC left, LabPi right).

If the recorded absorption maxima of the two solutions are compared, there are only minor differences in the absorbed wavelength of the purchased and synthesized nanoparticles per spectrometer. Using the dilution series, the concentration of the synthesized nanoparticles can now be determined **(Fig. 3)**.

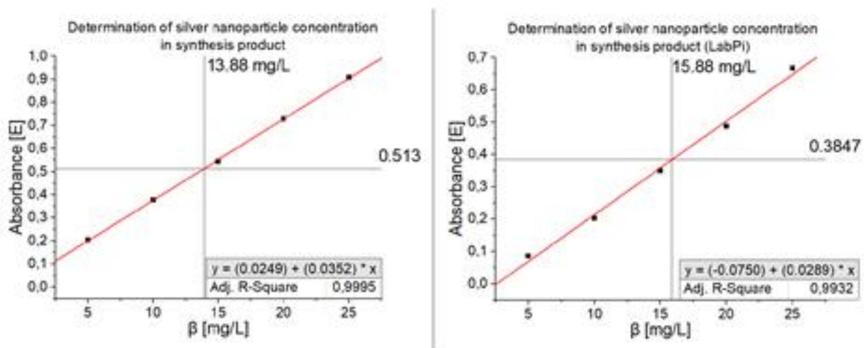

Fig. 3: Mass concentration of synthesized silver nanoparticles determined through photometric analysis (VWR Spectrophotometer UV-3100PC left, LabPi right).

Although it was not possible to achieve the possible yield of 22.88 mg/L, the assumption from the literature is sufficient to use the synthesized dispersion for further concentration series even without preceding content determination and to determine the mass concentration of silver nanoparticles in a cleaning agent. For this purpose, an absorption spectrum was used analogously to determine whether the size of the nanoparticles in the cleaner corresponded to the order of magnitude of approx. 50 nm (**Fig. 4**).

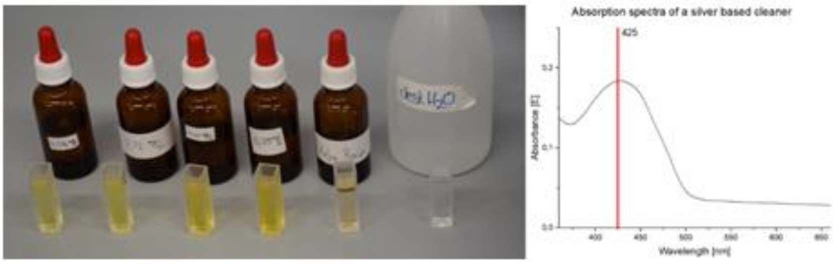

Fig. 4: Left: Dilution series and sample of the cleaner. Right: Recorded spectrum of the cleaner.

Based on the absorption maximum at 425 nm, it can be assumed that the nanoparticles are in the approximate size range and have a mainly spherical shape. With the aid of the dilution series from synthesized nanoparticles, the concentration of the silver nanoparticles in the cleaner can now also be approximated photometrically (**Fig. 5**).

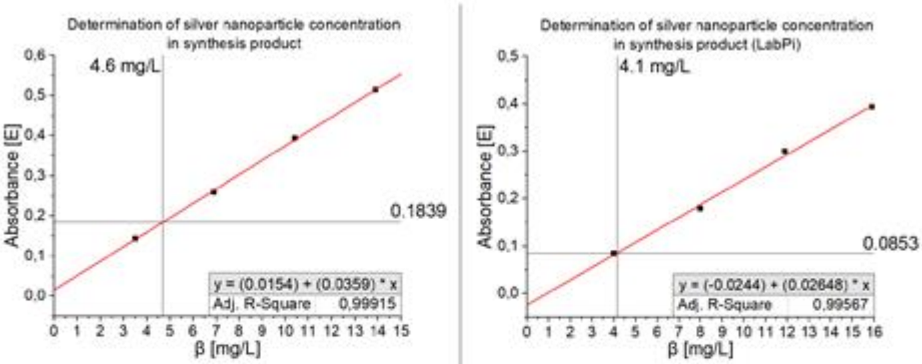

Fig. 5: Mass concentration of silver nanoparticles in cleanser determined through photometric analysis (VWR Spectrophotometer UV-3100PC left, LabPi right).

The most accurate approximation is made using purchased nanoparticles and measurement in the laboratory spectrometer, but similarly good results can also be obtained with the low-cost LabPi measuring station. The concentration of silver nanoparticles in the cleaner could thus be determined to be 4.6 mg/L (cf. LabPi: 4.1 mg/L).

It should be noted, however, that this series of experiments is only an approximation and a simplified observation for use in schools. The purchase of a laboratory spectrometer and the silver nanoparticles is often associated with high costs (100 mg nanoparticles ≈ 185 €), but the results shown here demonstrate that sufficiently good results can be obtained when analyzing silver nanoparticles using self-synthesized silver nanoparticles and working with low-cost equipment such as LabPi.

4. Practical experience

In a first pilot project with chemistry students, the students first synthesized silver nanoparticles and determined the concentration of nanoparticles in a cleaner photometrically with LabPi based on the theoretical values. In this process, the students showed interest in the silver nanoparticles, their properties, and applications. For photometry, they were able to rely on their prior knowledge in a targeted manner.

The experiments were then used as part of a summer school about nanotechnology. A first impression about spectroscopic methods was created among the students, but also the amount of nanoparticles in the cleaner could be determined. Suitable model experiments on opportunities and risks were contextualized here via other nanomaterials (*e.g.*, zinc oxide) [1].

Fig. 6: Pilot: Student measuring (left). Student synthesizing the nanoparticles (center). Student measuring with LabPi (right).

5. Outlook

The feedback received from the pilot provides promising initial insights. The experiments and the LabPi measuring station will now be revised accordingly for further work on the topic. Furthermore, additional everyday objects are to be developed in order to create additional points of contact to the students' everyday lives. Finally, the series of experiments will be expanded into a teaching sequence for K-12 school chemistry education.

References

[1] R. Saadat, B. Bartram, T. Wilke (2019): Made to Measure: Easy Synthesis and Characterization of Nanocomposites with Tailored Functionalities for School Chemistry Education. W. J. Chem. Ed., 2 (7), 65-71.

[2] R. Nonninger, J. Dege, T. Wilke, T. Waitz (2016): Nanoscience Education in School Chemistry: Perspectives for Curricular Innovations in Context of an Education for a Sustainable Development. In: K. Winkelmann und B. Bhushan (Eds.): Global Perspectives of Nanoscience and Engineering Education. Cham: Springer International Publishing, 237–274.

[3] M. Wejner, T. Wilke (2019): Low Cost – High Tech: Die digitale Messstation LabPi, CHEMKON, 26 (7), 294-300

[4] Leybold Didactics – Cassy. https://www.leybold-shop.com/technology/geraetekatalog-automotive/7x/cassy-automotive- measuring-set-738997.html (last visited: 19.01.2022)

[5] M. Wejner, T. Wilke (2022): LabPi: A digital measuring station for STEM Education 4.0. Journal of Chemical Education, DOI: 10.1021/acs.jchemed.1c01139

[6] S. Kittler, C. Greulich, J.S. Gebauer, J. Diendorf, L. Treuel, L. Ruiz, J.M. Gonzalez-Calbet, M. Vallet-Regi, R. Zellner, M. Köller, M. Epple (2010). The influence of proteins on the dispersability and cell-biological activity of silver nanoparticles. J. Mater. Chem. 20 (3), 512–518.

[7] H. Wang, X. Qiao, J. Chen, S. Ding (2005). Preparation of silver nanoparticles by chemical reduction method. Colloids Surf. A Physicochem. Eng. 256 (2), 111-115.

[8] J.N. Helmlinger (2015). Silber-Nanopartikel mit definierter Morphologie. Darstellung, Eigenschaften und biologische Wirkung. Dissertation. University Duisburg-Essen.