May 04, 2021

Synthesis and Characterization of Silver Nanoparticles using Sodium Borohydride as a Reducing Agent

Forked from Synthesis and Characterization of Silver Nanoparticles using Sodium Borohydride as a Reducing Agent

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1 Works for me dx.doi.org/10.17504/protocols.io.buq4nvyw

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ABSTRACT

Silver nanoparticles (AgNP) have a multitude of uses, which range from anti-microbial to anti-cancerous etc. They are sometimes preferred to other metal-based nanoparticles due to their attractive physicochemical properties. This protocol is a standardized method of producing and characterising PVA coated AgNPs which range from 10 to 20 nm in diameter. These particles are suited to lab based in-vivo and in-vitro assays. The addition of PVA to AgNPs is to increase the stability of the nanoparticles.

DOI

dx.doi.org/10.17504/protocols.io.buq4nvyw

PROTOCOL CITATION

Sean Byrne McDonnell, Sebnem Gunes, Alan Casey, James F Curtin, Eline Manaloto 2021. Synthesis and Characterization of Silver Nanoparticles using Sodium Borohydride as a Reducing Agent . **protocols.io** https://dx.doi.org/10.17504/protocols.io.buq4nvyw

FORK NOTE

FORK FROM

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KEYWORDS

Nanoparticles, SIlver, Toxicity , AgNP

CREATED

May 04, 2021

LAST MODIFIED

May 04, 2021

PROTOCOL INTEGER ID

49660

GUIDELINES

While performing **Step 5** ensure droplets are added into the vortex, avoiding contact with the walls of the beaker.

MATERIALS TEXT

- **30 mL** Sodium Borohydride (0.002M)

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- **2 mL** Silver Nitrite (0.001M)

- **2 mL** 1% Polyvinyl Alcohol in Millipore water (Simplicity 185, 18.2 MΩ.cm at 25°C resistivity)

- Ice bath
- Centrifuge (Capable of >5000 RPM)
- Stir Plate
- Magnetic stir bar
- Erlenmeyer flask (x2)
- Centrifugation tube (3kDa)
- Dropper
- Atomic Absorption Spectroscopy (Varian SpectrAA 200)
- Uv-Vis Spectrophotographer (Shimadzu, UV-1800)
- Dynamic Light Scattering Instrument (Malvern Instruments, UK)
- STEM Microscope (Hitachi SU 6600)

SAFETY WARNINGS

PPE must be worn when handling sodium borohydride, as it poses the following risks.

- H260: In contact with water releases flammable gases which may ignite spontaneously [Danger Substances and mixtures which in contact with water, emit flammable gases]
- H301: Toxic if swallowed [Danger Acute toxicity, oral]
- H311: Toxic in contact with skin [Danger Acute toxicity, dermal]
- H314: Causes severe skin burns and eye damage [Danger Skin corrosion/irritation]
- H318: Causes serious eye damage [DangerSerious eye damage/eye irritation]
- H332: Harmful if inhaled [WarningAcute toxicity, inhalation]
- H360: May damage fertility or the unborn child [Danger Reproductive toxicity]

BEFORE STARTING

Before starting the process, set up an ice bath that contains a magnetic stir plate. This will be used throughout the synthesis process.

Fresh stock solutions of both 0.002M $NaBH_4$ and 0.001M $AgNO_3$ should be prepared before starting each time.

For PVA-AgNP concentration measurements (AAS) prepare 1, 2, 3, 4, 5 µg Ag/ml calibration standards using a stock soloution of 1g/L AgNP in millipore water.

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Synthesis

1 Sodium Borohydride **Sigma** **Aldrich Catalog #213462-25G**

Add **30 mL** of **0.002 Molarity (M)** to an Erlenmeyer flask.

2 20m

Insert the Erlenmeyer flask containing **30 mL** of sodium borohydride into the previously set up ice bath containing the stir plate and allow to cool for **00:20:00**

3 Place a magnetic mixing rod into the Erlenmeyer flask and start the stir plate to initiate mixing.

4 In another Erlenmeyer flask, transfer **2 mL** of the freshly prepared Silver Nitrite **Sigma** **Aldrich Catalog #209139-25G** along with **2 mL** of a 1% PVA solution.

1% Polyvinyl Alcohol in Millipore water (Simplicity 185, 18.2 MΩ.cm at 25°C resistivity)

5 At a rate of one drop per second, drip **2 mL** of the AgNO3/PVA solution into the chilled Sodium Borohydride **Sigma** **Aldrich Catalog #213462-25G** solution, maintaining the cooling and mixing simultaneously.

6 When the AgNO3/PVA solution has been added to the Sodium Borohydride **Sigma** **Aldrich Catalog #213462-25G** solution, stop mixing immediately.

Once the reaction has been stopped, note that a yellow solution should be formed. This indicates that PVA-AgNP formation has occurred.

Purification

7 Transfer **20 mL** of the PVA-AgNP solution, along with the excess Sodium Borohydride **Sigma** **Aldrich Catalog #213462-25G** into an ultrafiltration tube of 3kDa.

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3kDa Ultrafiltration tube used in this protocol was from Sartorius.

This step may need to be repeated for volumes >20mL.

8 Transfer the tube into a centrifuge and run at **5000 rpm, Room temperature , 00:40:00**

9 Post centrifugation, smaller NPs (>5µm in diameter) and excess water (including unwanted reactants) can be discarded from the ultrafiltration tube.

The smaller AgNPs will be seen at the clustered at the bottom of the tube.

10 The PVA-AgNP concentrate will display a dark brown colour, this is then collected using a dropper.

11 The harvested AgNPs must then be stored at fridge temperature (Approx. 4°C) away from direct sunlight.

Characterisation

12 AgNP samples are prepared using a 1 in 10 dilution by adding 1 ml concentrated PVA-AgNP to 9 mL of millipore water

13 For primary characterisation, **3.5 mL** of PVA-AgNP's are placed into a cuvette, a UV-Vis spectrum is ran from 300nm-600nm.

Strong absorbance at 400nm indicates 10mm AgNP synthesis.

14 Using a DLS instrument (Malvern Instruments, UK) particle size range is measured by placing **3.5 mL** PVA-AgNP soloution into a 12mm o.d. square polystyrene cuvette and ran as per manufactuer specification.

DLS Measures hydrodynamic size, including both the PVA coating the AgNP's.

Zetasizer software was used to calculate the size distribution.

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15 The DLS instrument was again used to measure the Zeta Potential of the AgNPs, this was performed on the same sample specifications for the previous DLS sample .

Zeta potential measures the potential for the particles to flocculate, with a high negative or positive charge causing them to repel one another.

16 STEM Characterization, **3 µl** of PVA-AgNP aqueous solution is placed onto a carbon-coated copper plate and allowed to dry. Particles are then analyzed visually for dispersion and shape.

Drying allows for the highest yield of particles on the grid.

17 Using the previously prepared calibration solutions (1-5 ug AgNP/mL) the concentration of the prepared AgNP solution was calculated using AAS (Varian SpectrAA 200).

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