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A domestic pressure cooker mediated, facile autoclaving method for the synthesis of silver nanoparticles

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ABSTRACT

As an alternative to an expensive hydrothermal reactor, in the current method a domestic pressure was used for the synthesis of silver nanoparticles (Ag NP) using tree gum, kondagogu as dual functional reductant and stabilizer by autoclaving. The formation of Ag NP was evaluated with colour transformation, UV-Visible spectroscopy (UV–Vis) and transmission electron microscopy. The formation of Ag NP by gum confirmed from the developed yellow coloration of the solution and the appearance of surface plasmon resonance peak at 408 nm in the UV–Vis. The produced NP were spherical, polydisperse, particle size ranged from 2.9–17.6 nm and the average particle size was 4.5 ± 3.1 nm. The developed method is useful for demonstration, gaining hands on experience and production of metal and metal oxide NP in resource limited small laboratories, rural colleges, startups etc.

- Pressure cooker serves as an easily accessible, durable, inexpensive, electricity independent hydrothermal nanoparticle production vessel.
- Autoclaving serves as a facile, ecofriendly, less energy consuming, one pot, green method with dual functional role of *in situ* nanoparticle synthesis and sterilization.
- Production of intrinsically safe and sterile nanoparticles amenable for *in vivo* and *in vitro* biomedical applications.

Specifications table

Subject area:	Materials Science
More specific subject area:	Facile, nanoparticle synthesis using domestic pressure cooker
Name of your method:	A domestic pressure cooker mediated, facile autoclaving method for the synthesis of silver nanoparticles
Name and reference of original method:	A.J. Kora, R.B. Sashidhar, J. Arunachalam, Gum kondagogu (*Cochlospermum gossypium*): A template for the green synthesis and stabilization of silver nanoparticles with antibacterial application, Carbohydrate Polymers 82(3) (2010) 670-679, doi:10.1016/j.carbpol.2010.05.034
Resource availability:	Not applicable

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Method details

Background

Among the processes of nanoparticle production, hydrothermal methods that result in nanoparticle formation at elevated temperature (> 25 ºC) and pressure (> 100 kPa) in an aqueous medium gained attention. It is mainly due to the generation of nanoparticles with superior purity and stoichiometry; tunable particle size and shape; narrow particle size distribution, rapid commercial production in bulk scale in batch or continuous mode, cleaner methodologies, elimination of the requirement of elevated temperature calcination and milling etc [1]. The hydrothermal synthesis is generally carried out with a specialized apparatus that serves as a hydrothermal reactor, such as high pressure bearing autoclave made up of stainless steel (SS) or high strength alloy with Teflon or polytetrafluoroethylene lining [2,3]. The synthesis is optimized by varying temperature, reactant concentrations, duration etc [1,4]. Also, the autoclave apparatus is most commonly used for sterilizing the culture media, plasticware, glassware and decontaminating the used labware, grown microbial cultures etc in which the microbes are killed under high steam pressure at 121˚C within 20–30 min, based on the loaded contents [5]. However, in resource constrained conditions [4], small laboratories, rural colleges and especially in countries like India; it is commonly replaced with a domestic stovetop pressure cooker.

After the introduction, the usage of pressure cookers gained momentum and was popularized in 1980 and they became an essential kitchen appliance of every Indian kitchen and home due to the enhanced usage of liquefied petroleum gas (LPG) as a result of government policies. It has in built safety features such as a controlled gasket release system, overpressure metallic safety plug and pressure regulator/precision weight valve and is available in various models based on the features such as on the type of lid (inner, outer), material (aluminium, hard anodized, SS), vessel shape (straight wall, pan), base (induction, non induction) and capacity (1.5–20 L) [6]. The revolutionary, durable household pressure cooker is both LPG and induction stovetop compatible and does not mandate electricity for operation. For example, a 12 L capacity, straight walled, aluminium, dual top compatible pressure cooker with 5 years warranty costs around USD 50, which is much cheaper than the electric pressure cooker (USD 100) and hydrothermal autoclave (USD 3000) [4].

Thus, in the current method a domestic pressure cooker was used as an inexpensive hydrothermal reactor for synthesizing silver nanoparticles (Ag NP) using tree gum, kondagogu as dual functional reductant and stabilizer. The formation of Ag NP was evaluated with colour transformation, UV-Visible spectroscopy (UV–Vis), X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), dynamic light scattering (DLS) and transmission electron microscopy (TEM).

Materials and methods

Gum kondagogu (Girijan Co-operative Corporation, Hyderabad, India), silver nitrate ($AgNO_3$) (Merck, Mumbai, India) and ultrapure water (Elga, High Wycombe, England) were used during the Ag NP synthesis. A 0.5 % gum solution in ultrapure water was prepared from 38 μm sized gum powder by overnight stirring at room temperature and 100 mM stock solutions of $AgNO_3$ was prepared in ultrapure water and aliquoted into lightproof small vials (Fig. 1). A 12 L volume, non induction base, LPG stovetop (TTK Prestige, Bengaluru, India) compatible, aluminium pressure cooker (TTK Prestige, Bengaluru, India) was used as a sealed hydrothermal reactor for NP synthesis (Fig. 2) [7,8]. The Ag NP were prepared at optimized condition of 0.5 % gum and 1 mM of $AgNO_3$ at a reaction time of 60 min using the domestic pressure cooker (Fig. 3). The synthesis was standardized by varying the gum and $AgNO_3$ concentrations and time of autoclaving [9].

Steps during the synthesis

The pressure cooker used in the current protocol was filled with one litre of water to maintain a minimum water level of 1 inch. Then, the loosely capped glass bottles containing the reaction mixtures of gum and $AgNO_3$ were placed in a water filled glass beaker in which the water level should be more than the bottle solution level. After placing the bottles containing the beaker in the cooker, the lid closed cooker was placed on the LPG stove and the burner was ignited at high flame. The opening of the steam tube/vent pipe was closed with the pressure regulator weight valve, once the cooker was filled with steam and ejected/escaped through the vent opening. After getting/hearing the whistle sound from the weight valve, the flame was reduced to a simmer level and started counting the reaction/autoclaving time. After 60 min of autoclaving the flame was turned/switched off, removed the cooker from the stovetop carefully, placed on a granite platform, allowed it to cool and reach the ambient temperature. Once the cooker was cooled and the pressure inside was reduced, the beaker containing the bottles was taken out and the bottle solutions were checked for synthesized NP. The formation of nanoparticles by autoclaving was checked by solution colour change, recording the UV–Vis spectra (Analytik Jena AG, Jena, Germany), XRD pattern (Rigaku, Tokyo, Japan), IR spectra (Bruker Optics, Ettlingen, Germany), z average and zeta potential (Malvern, Malvern, UK) and capturing the TEM (FEI, Eindhoven, Netherlands) images.

Precautions

The critical points and precautions to be taken care of during the pressure cooker mediated NP synthesis is explained here.

(1) Dispersion of solutes and mixtures:

The reducing/stabilizing agents used for the NP synthesis should be entirely dispersed in the aqueous medium for efficient synthesis, especially the viscous tree gums. It can be achieved with overnight stirring during gum stock preparation and 30 min prior stirring to synthesis. The frozen $AgNO_3$ stock vial should be thawed to room temperature and mixed vigorously before using it for NP

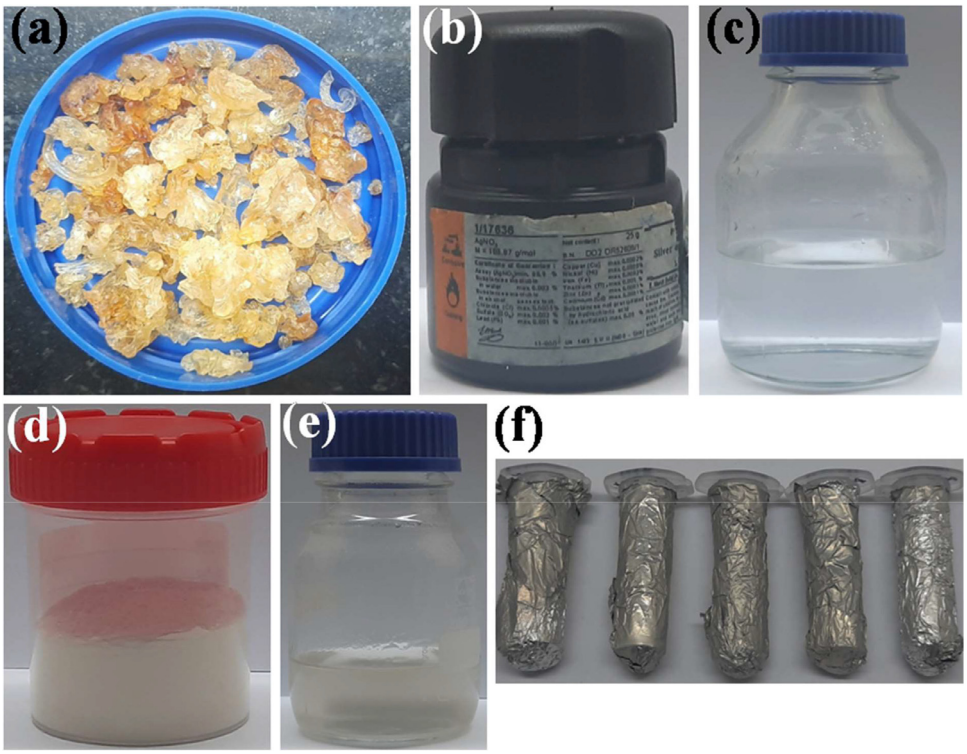

Fig. 1. Photographs of the materials used for the Ag NP synthesis by pressure cooker autoclaving, (a) gum kondagogu, (b) silver nitrate (c) ultrapure water, (d) powdered gum, (e) 0.5 % gum solution and (d) 100 mM stock solutions of silver nitrate in small vials.

synthesis. After mixing the bioreductant and metal precursors, the solution should be thoroughly mixed for efficient synthesis and prevention of gradation/gradient formation.

(2) Stock solution preparation and storage:

It is preferable to prepare the bioreductant stock solutions in sterile containers using sterile ultrapure water and can be further filter sterilized to prevent bacterial and fungal contamination, as they are rich sources of sugars, proteins and minerals. The sterile bioreductant stock solutions should be stored at 4 ºC to enhance the shelf life further. If any contamination occurs in the bioreductant stock, it should be discarded. Also, the stock solutions of expensive metal precursors such as $AgNO_3$ should be prepared at a higher concentration of 100 mM in a volume of 10 mL, not at a large volume (100 mL). The stock solution should be aliquoted into smaller volume vials (1–2 mL) and stored at -20 ºC either by wrapping with aluminium foil or in light tight/dark vials. It will be helpful in the prevention of silver ion photooxidation, evaporation induced volume loss and concentration difference; and cross contamination of expensive stocks with reductants during pipetting and transfers. If any black or yellow colouration of the stock occurs, that single vial can be discarded, not the entire vial stock. In addition, for the synthesis of 5 mL of 1 mM concentrated Ag NP solution, just 50 µL of 100 mM $AgNO_3$ stock solution is sufficient and it neither enhances the solution volume nor dilutes the bioreductant concentration during synthesis.

(3) Water level:

The water level in the pressure should be adequate (1 inch height) for longer durations of autoclaving. Also, the glass beaker kept inside the cooker should not float on the water surface and excessive water level both in the cooker and beaker can tumble the beaker and bottles, respectively. The water level in the beaker should be slightly higher than the bottle solution level for better heat transfer. The cooker and beaker should not be completely water filled to prevent the water dilution of the synthesized NP in loosely capped bottles during synthesis.

(4) Pressure built up:

The bottles used for NP synthesis should be loosely capped to prevent pressure building up inside the bottles and subsequent breakage. Also, the cooker should not be entirely filled with contents for the safer escape of developed steam through the vent pipe during and after autoclaving. Also, the cooker lid should be opened only after reaching the temperature and pressure inside the cooker to ambient conditions to avoid steam burns and explosions.

Fig. 2. Photographs of the apparatus and accessories used for the Ag NP synthesis, (a) LPG stove, (b) LPG cylinder and (c) domestic, LPG stovetop pressure cooker.

(5) Pressure cooker operation:

The necessary safety precautions needed for pressure cooker operation should be followed. In addition, the lid, vent pipe, pressure regulator valve, safety plug and gasket should be checked for leaks and clogged organic residues. After usage, the cooker should not be stored with the leftover water to prevent scale formation and for ensuring gas saving and efficient energy transfer for the successive cycles. Also, it should be regularly cleaned with mild detergent and stored/keep it dry for the upcoming cycles.

(6) LPG operation:

The essential and general safety precautions needed for using the LPG cylinder and stove such as fixing/keeping/storing the cylinder in an erect position at ground level on a flat surface, usage of safety standard certified leak proof cylinder valve, regulator and rubber tubing, regular service checks; using the stove it in a well ventilated area above the cylinder level on a flat slab, away from flammable materials, closing off the burner switch and cylinder regulator after use, regular cleaning of the clogged stove burners etc should be followed while using it. Neither the LPG stove nor the pressure cooker should be left unattended/unsupervised during the operation.

Method validation

Among an array of hydrothermal methods, household pressure cooker mediated autoclaving was utilized as a facile method for synthesizing Ag NP using tree gum kondagogu at standardized conditions. The readily available domestic pressure cooker functions at lower temperature and pressure limits and serves as a reaction vessel for cheaper production of nanomaterials [4]. The synthesis of Ag NP was evaluated with colour transformation, UV–Vis and TEM. The formation of Ag NP by tree gum via autoclaving was confirmed from the visual transformation of the solution colour into yellow (Fig. 4(a)) [9]. The UV–Vis absorption spectrum of the gum synthesized NP showed an absorption maximum at 408 nm, attributed to the charactestic surface plasmon resonance (SPR) peak of Ag NP (Fig. 4(b)) [7]. The XRD pattern exhibited four distinctive peaks at 38.2, 44.9, 64.6 and 78.1 º corresponding to (111), (200), (220) and (311) planes of face centered cubic crystal structure of elemental silver (Fig. 5) [10]. The FTIR spectra of gum and gum

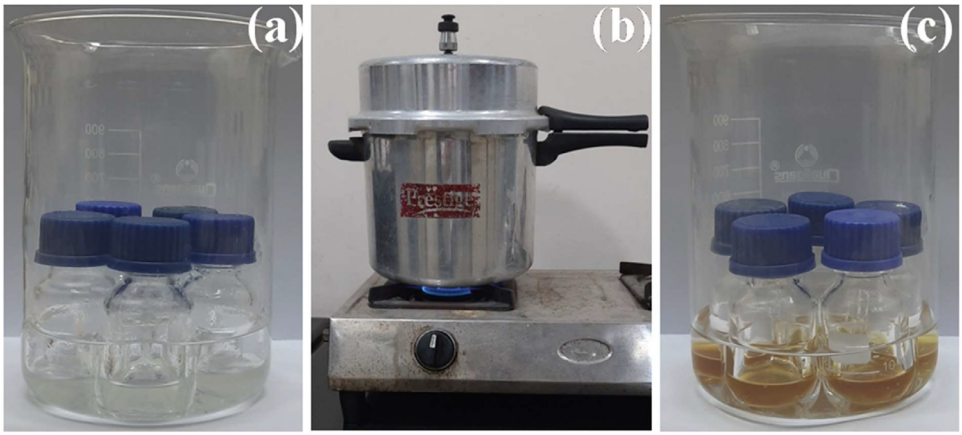

Fig. 3. Photographs of the steps involved silver nanoparticle synthesis by pressure cooker autoclaving, (a) beaker containing glass bottles with solutions of 0.5 % gum and 1 mM of $AgNO_3$, (b) pressure cooker mediated autoclaving and (c) synthesized Ag NP solutions.

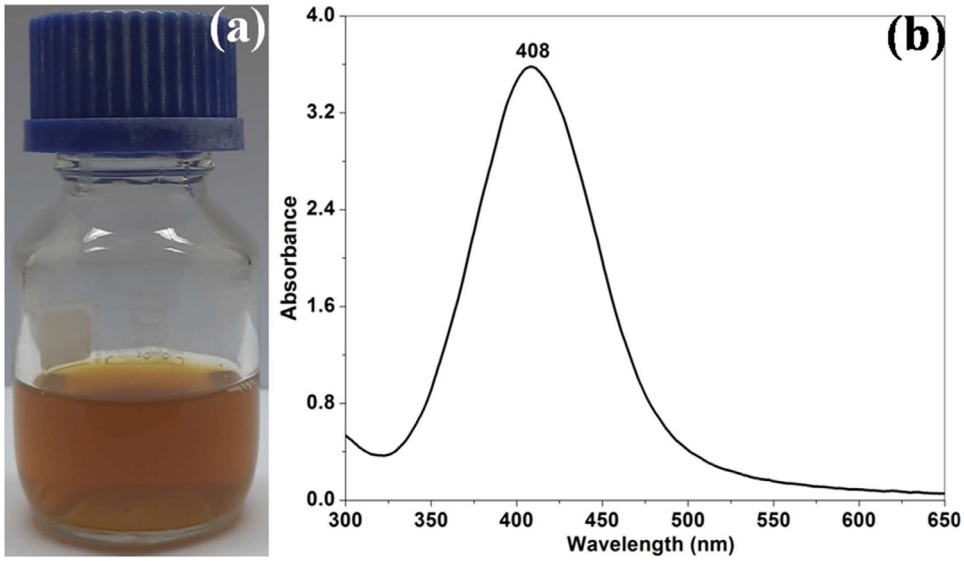

Fig. 4. (a) Yellow coloured Ag NP solution and (b) the UV–Vis absorption spectrum of the gum synthesized Ag NP solution.

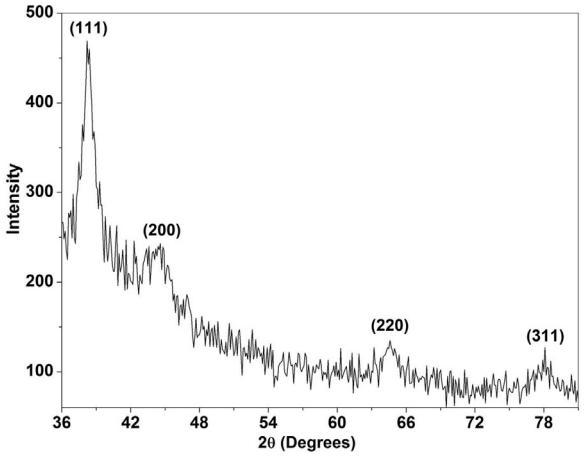

Fig. 5. The XRD pattern of silver nanoparticles showing face centered cubic crystal structure.

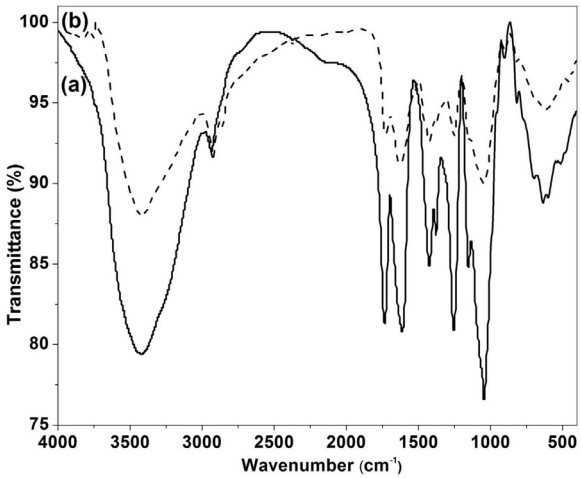

Fig. 6. The FTIR spectra of (a) gum and (b) gum produced silver nanoparticles.

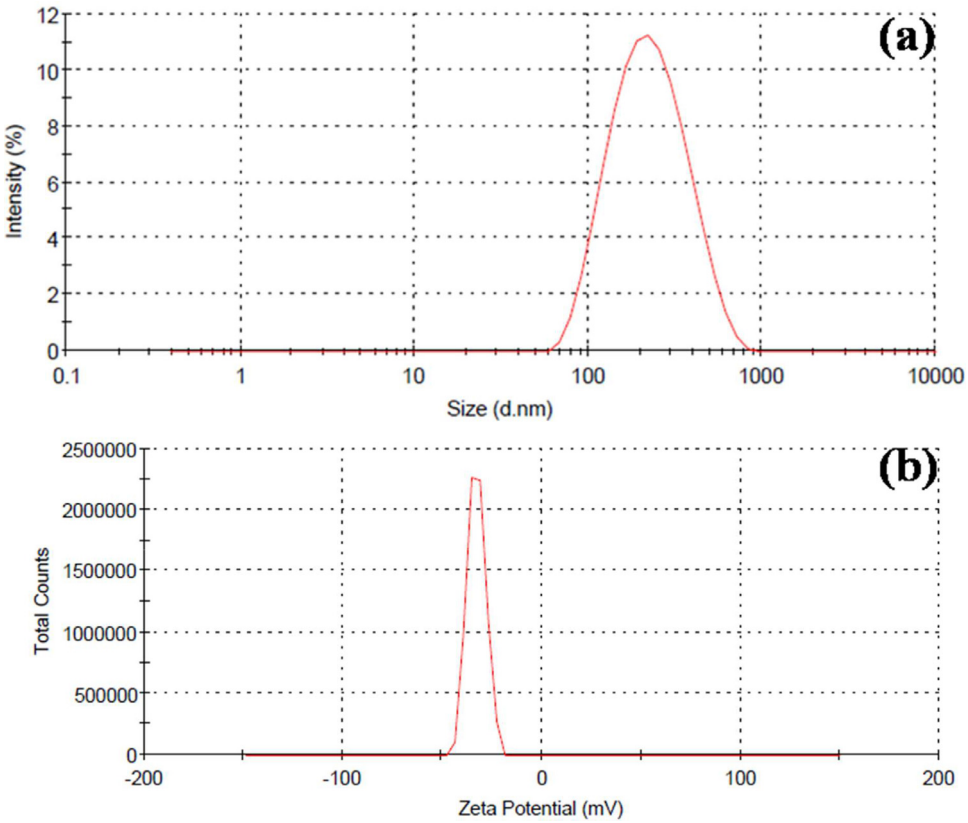

Fig. 7. The (a) particle size distribution and (b) zeta potential of synthesized silver nanoparticles.

produced NP indicated the involvement of hydroxyl and carbonyl groups; and proteins in the silver ion reduction and stabilization of NP, respectively (Fig. 6) [7–9,11]. The z average and zeta potential values obtained from DLS were 246 ± 16.4 nm and - 30.3 ± 3.1 mV, respectively confirming the nanoparticle capping and stabilization by gum molecules (Fig. 7) [12]. The TEM image of the produced NP was given in Fig. 8. The NP were spherical, polydisperse, particle size ranged from 2.9–17.6 nm and the average particle size was 4.5 ± 3.1 nm [9].

The advantages of the domestic pressure cooker mediated autoclaving method for the NP synthesis are, (i) the functioning of the pressure cooker as an easily accessible, durable, inexpensive, electricity independent hydrothermal nanoparticle production vessel with inbuilt safety features [4], (ii) utilization of environmentally and biologically benign, universal solvent water as a synthetic

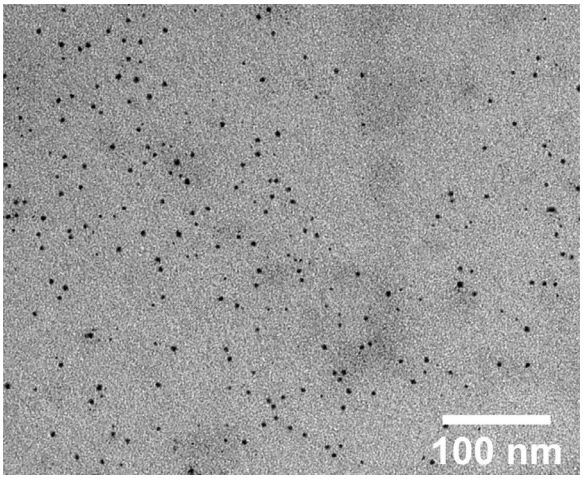

Fig. 8. The TEM image of the produced Ag NP at 100 nm scale.

medium [13], (iii) serving as a facile, ecofriendly, less energy consuming/energy efficient, one pot, green method for nanoparticle synthesis [9,13], (iv) efficient production of stable, non aggregated nanoparticles [7], (v) production of intrinsically safe and sterile nanoparticles amenable for *in vivo* and *in vitro* biomedical applications [5,7,14] and (vi) dual functional role of autoclaving towards *in situ* synthesis and sterilization [5]. Employing the same protocol, the Ag NP were synthesized from tree gums such as ghatti [15], olibanum [16], tragacanth [17]; bacterial gum xanthan [10], seed extract [11], leaf extract [7,18] etc. The developed method was exploited for the synthesis of other metal and metal oxide nanoparticles, such as selenium [19], palladium [12], platinum [8] and copper oxide [20].

Conclusion

The household pressure cooker mediated, hydrothermal autoclaving serves as a simple, one pot, cheaper method for the simultaneous synthesis and sterilization of Ag NP in a single step. The easily available soft hydrothermal reactor vessel is exceptionally beneficial for practically demonstrating the nanoparticle synthesis for students in rural colleges, gaining hands on experience in NP synthesis and optimization; and producing nanoparticles in resource limited small laboratories, startups, farmer cooperative societies etc. The pressure cooker synthesized Ag NP finds its application as a bactericide [7,9], fungicide [18], wound dressing, catalyst, colorimetric mercury sensor etc.

Declaration of Competing Interest

The author declares that she has no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgments

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