Novel Green Synthesis of Gold Nanoparticles: Facilitation of Synthesis of Colloidal Gold Nanoparticles Using Various *Citrullus lanatus* Extracts

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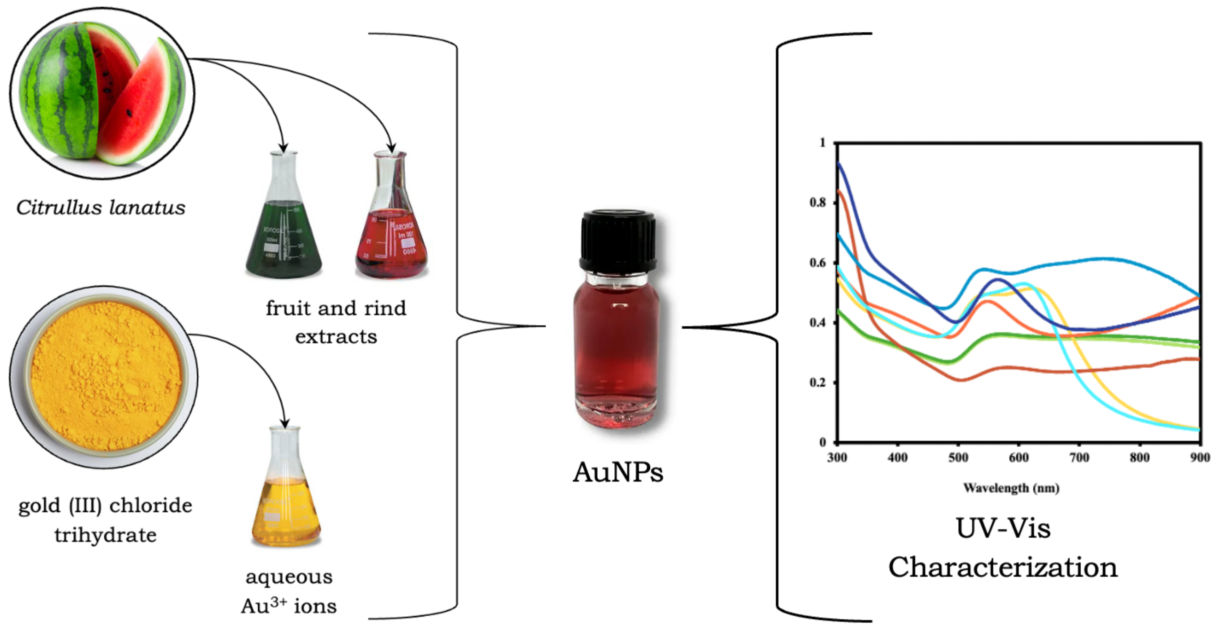
ABSTRACT

Gold nanoparticles (AuNPs) are a form of nanotechnology that retain size-dependent physical, chemical, and biological characteristics in comparison to their macroscopic source material, with useful applications in medicine, electronics, and environmental management through exploitation of their surface area and present active sites. As traditional methods of AuNP synthesis incur high energy demands and environmental risks from the use of hazardous reagents, identification of green synthesis methods retains significant interest. This study presents the novel synthesis of AuNPs via a green synthesis method, applying aqueous solutions of watermelon rind and fruit extracts, with subsequent UV-Vis spectroscopic characterization. Synthesized AuNPs typically displayed absorbance in the 520-540 nm range, characteristic of nanoparticles 12-41 nm in diameter, while some variance, specifically displayed by AuNPs synthesized using rind-sourced extracts, may indicate variance in size and shape. Variation in volumetric ratio of experimental reagents also highlighted manipulation of such ratios as a possible useful tool in controlling aggregation and subsequent size of colloidal gold particles. While this study affirms the efficacy of the synthesis method, further determination of both the exact size and shape of nanoparticles utilizing TEM (transmission electron microscope) imaging as well as characterization of watermelon extracts by way of FT-IR (Fourier-transform infrared) spectroscopy to identify specific chemical agents responsible for formation of AuNPs may be valuable.

GRAPHICAL ABSTRACT



KEYWORDS

first-year undergraduate, second-year undergraduate, upper-division undergraduate, research, analytical chemistry, organic chemistry, inorganic chemistry, interdisciplinary, analogies/transfer, inquiry-based, problem solving, aqueous solution chemistry, colloids, green chemistry, materials science, nanotechnology, qualitative analysis,spectroscopy,UV-vis spectroscopy, water chemistry

1. INTRODUCTION

Gold nanoparticles (AuNPs) have shown capacity for diverse applications, ranging from medical and biochemical contexts such as drug delivery and medical imaging, to novel applications in photonics and spintronics.[1,2] AuNPs retain unique characteristics distinct from those found in their macroscopic source materials, such as high surface area and active sites that facilitate reductive and oxidative reactions, as well as ease of functionalization with biomolecules such as proteins or DNA.[2] Application of AuNPs in biomedical and pharmaceutical contexts necessitates their production on a large-scale through non-toxic methods, with application of plant extracts, yeasts, fungi, and other materials emerging as valuable alternatives to traditional methods of synthesis[3]. Specifically, nanoparticles produced via green synthesis methods applying plant extracts are particularly stable, with increased formation rates in comparison to nanoparticles formed utilizing alternative biological means.[2]

Watermelon (*Citrullus lanatus*) is a widely consumed and produced fruit, with a composition including compounds such as cellulose, citrulline, pectin, proteins, and carotenoids[2]. Previous research has shown that once extracted, these compounds can facilitate the formation of gold nanoparticles by acting as reducing and capping agents[2,3,4]. When introduced to gold ions in solution, Au^{3+} ions are bio-reduced to their metallic form, initiating nanoparticles nucleation, with smaller particles progressively clustering together to yield more thermodynamically stable nanoparticles.[2] As nanoparticles retain unique properties depending on their size and shape, UV-Vis spectroscopy becomes a useful tool for characterization, with specific sizes and shapes of nanoparticles typically retaining their own respective optical properties, and thereby wavelengths of maximum absorption.

In this study, it was aimed first to determine a suitable method for preparation of watermelon fruit and rind extract, before verifying the efficacy of a green synthesis using such extracts in facilitating nanoparticle formation. Additionally, variation in volumetric ratio of reagents was implemented to investigate the impact on formed nanoparticles. Synthesized AuNPs were finally characterized using UV-Vis spectroscopy, to both verify their presence as well as compare consistency of optical properties between AuNPs synthesized by different reaction mixtures.

2. SYNTHESIS OF GOLD NANOPARTICLES

2.1 Materials and Instrumentation

Gold (III) chloride trihydrate (99.9% purity) was utilized to prepare Au^{3+} ion solution. Gold salt was provided by Sigma Aldrich and was not purified before use. Prior to characterization, a Branson 1800 Cleaner was utilized to sonicate samples, before their processing with a Cary 60 UV-Vis spectrometer. Gold fruit and rind was sourced from local supermarkets in the Hampton, VA area.

2.2 Preparation of *Citrullus lanatus* Extracts

After obtaining watermelon fruit in wedged slices, watermelon fruit was separated from the rind, with the rind being considered as encompassing both the outer skin as well as light green layer beneath the fruit. Watermelon fruit was used for the preparation of red extract (RE) while rind was used to produce green extract (GE) (**Fig. 1**). To prepare both extracts, 40 g each of cubed fruit and rind was added to two separate glass beakers and combined with approximately 200 mL of deionized water. A simple extraction was performed by bringing both mixtures to a boil for fifteen minutes, before allowing mixtures to cool and performing a dual separation of crude extracts with the employment of both a sieve as well as a separatory funnel employing Whatman Grade 1 filter paper for the removal of bulky residues. Resulting extracts were kept refrigerated when not in use.

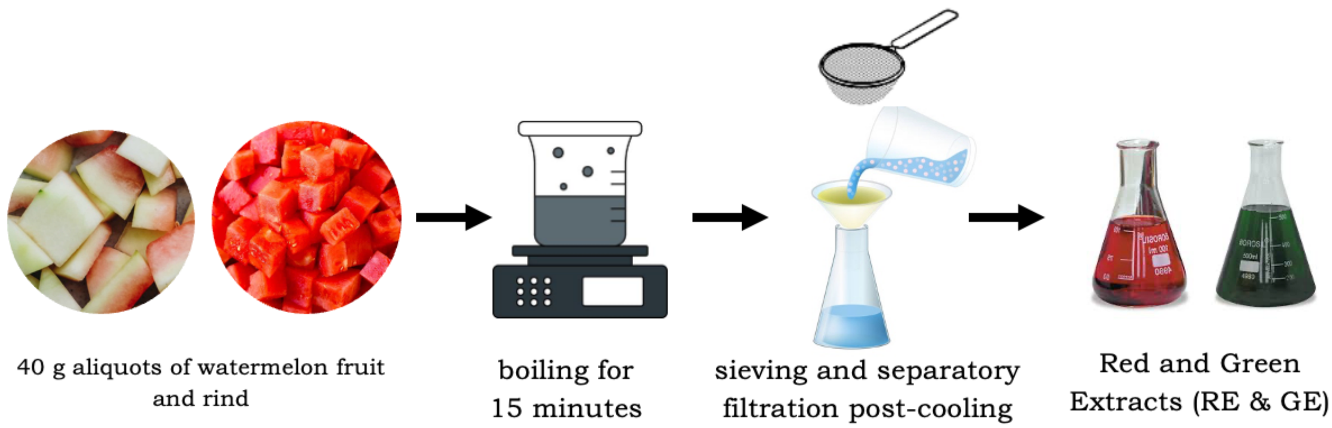


Figure 1: Stepwise schematic diagram for the simple extraction of watermelon fruit and rind to yield red and green watermelon extracts (RE & GE).

2.3 Preparation of Aqueous Solutions

To prepare a 1.27 mM gold ion solution, 6.8 mg of gold (III) chloride trihydrate (99.9% purity) was combined with 12 mL of deionized water in a resealable 20 mL glass vial, which was covered when not in use. Desired 15% by volume concentrations of both red and green extracts were prepared by adding 3.75 mL each of both extracts to two separate 25 mL volumetric flasks, subsequently adding 21.25 mL of deionized water to each and swirling to mix.

2.4 Preparation of AuNP Reaction Mixtures

Nanoparticle synthesis using red and green extracts was facilitated identically, with variation in experimental reagents being applied identically. Varying amounts of aqueous extracts (5, 4.5 and 3 mL each) were combined with varying amounts of Au^{3+} ion solution (1, 1.5 and 3 mL respectively) to form 5:1, 3:1, and 1:1 aqueous extract to ion solution reaction mixtures. Reagents were combined in glass

10 mL test tubes, and the reactions were allowed to proceed unagitated at room temperature while covered with parafilm.

2.5 UV-Vis Characterization

To prepare samples for characterization, reaction mixtures were first sonicated in a Branson 1800 cleaner until all present precipitate was observed as either dissolving or becoming suspended in solution. 2 mL of each reaction mixture was diluted with 1 mL of water in a 3 mL spectrometer cuvette. A Cary 60 UV-Vis spectrometer operating at room temperature was used to produce absorption spectra over a range of 300-800 nm. Resulting data was processed and analyzed in Microsoft Excel.

3. DATA AND RESULTS

Formation of AuNPs was monitored using UV-Vis Spectroscopy, with colloidal solutions displaying maximum absorption in the mid to late 500 nm range when monitored across a range of 300-800 nm. Relative consistency between wavelengths of maximum absorption was observed within 10-20 nm between solutions (**Fig.3**). The highest values of absorbance were displayed by reaction mixtures featuring the use of 15% by volume red extract, with solutions prepared with green extract typically yielding bother lower absorbances as well as more right-shifted wavelengths of maximum absorbance. For both extracts, relative maximum wavelength increased with the decrease in volumetric ratio of extract to gold solution, indicating a possible relationship between volumetric ratio and resulting size or shape of synthesized nanoparticles (**Table 1**).

Reaction mixtures remained colorless when first prepared, with 5:1 and 3:1 reaction mixtures obtaining a violet-red yet transparent appearance after 24 hours, which deepened and displayed lessened transparency after seven days, and even more so after sonication (**Fig. 2**). Contrastingly, 1:1 reaction mixtures retained bronze-yellow coloring after 24 hours but followed patterns of deepening and lessened transparency after seven days and post sonication (**Fig. 2**).

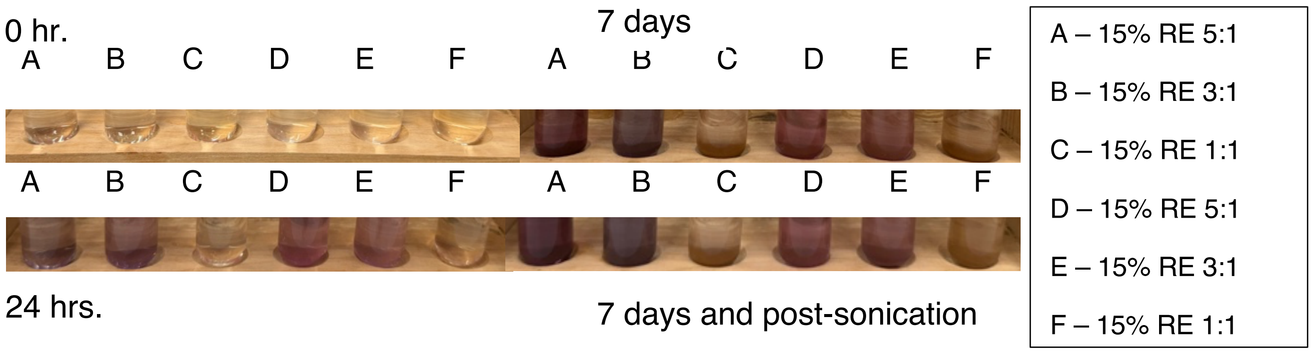


Figure 2: AuNP Reaction mixtures 0 hr., 24 hrs., and seven days post-synthesis (pre- and post- sonication)

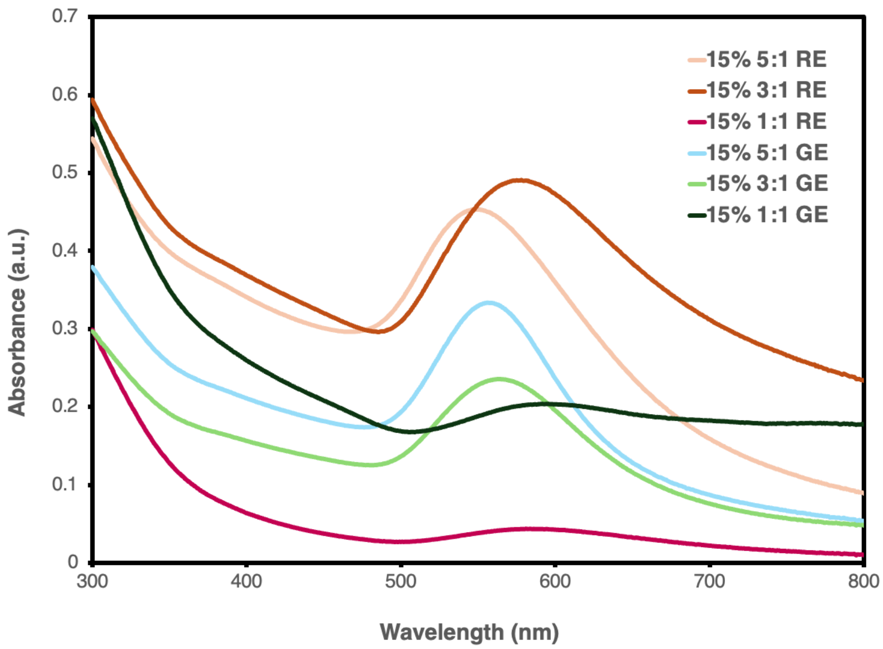


Figure 3: UV-Vis absorption spectra of colloidal gold reaction mixtures containing aqueous AuNPs, obtained by processing in Cary 60 spectrometer across a range of 300-800 nm.

Table 1: Relative wavelengths of maximum absorption in the 500-600 nm range from synthesized AuNP reaction mixtures.

Au NP Reaction Mixture	Relative λ_{max} (500-650 nm range)
15% RE:Au^{3+} Solutions (vol. ratio)	
5:1	547
3:1	578
1:1	584

15% GE:Au^{3+} Solutions (vol. ratio)	
5:1	556
3:1	564
1:1	597

4. CONCLUSIONS

In the present study, aqueous extracts derived from watermelon fruit and rind were evaluated for their efficacy in successfully facilitating the reduction of Au^{3+} ions into stable AuNPs. Formation of AuNPs was verified by UV-Vis spectroscopy, revealing maximum absorption in the mid to late 500 nm range, consistent with colloidal gold solutions, albeit with caveats and variation depending on size and shape of AuNPs.[5.] Results verify the capacity or watermelon-derived extracts to result in the formation of colloidal gold solutions, but do not identify the specific compounds responsible for the reducing and capping of aqueous gold ions, or the size and shape of produced nanoparticles.

Future Directions

In the future, further characterization of watermelon extracts may be useful in determining what specific compounds contribute to the formation of AuNPs and the development of a concise reaction mechanism, which could be achieved by way of both UV-Vis spectrometric as well as FT-IR (Fourier-transform infrared) spectroscopic characterization of watermelon extracts post extraction. Additionally, further characterization of the size and shape of AuNPs could be determined by application of TEM (transmission electron microscope) imaging, with further analysis of controlling parameters of size and shape being achieved by implementing time-course focused repetition of the experimental procedure. The impacts of an alternative method of extracts, such as hydrodistillation or drying or source material, as well as the implementation of heating or agitation of reaction mixtures during synthesis may also be investigated.

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ACKNOWLEDGMENTS

I would like to thank Dr. Peter Njoki, as well as Hampton University's Department of Chemistry and Biochemistry at large for providing both the resources and environment necessary to facilitate this research.

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