

# Making Nanoparticles Mega: Scaling-up the Synthesis of Gold Nanoparticle-Wool Fibre Composites

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## ABSTRACT

Due to their intense surface plasmon resonances in the visible region of light, gold nanoparticles have potential as attractive, colourfast dyes for premium wool fibres. Gold nanoparticle-wool fibre composite materials may be fabricated into luxury apparel, carpet and textiles for international markets. These proprietary novel hybrid materials which are being commercialised by Noble Bond Ltd have stimulated immense international interest and demand, desiring demonstration products of significant sizes for market appraisal. The synthesis of gold colloids is easily reproducible on the laboratory-scale, however, significant difficulties arise when large volumes of gold nanoparticles are required. This paper presents a non-technical overview of the synthesis of gold nanoparticle-wool fibre composites going from the laboratory-scale to the multiple kilogram-scale.

**Keywords:** gold, nanoparticles, wool, scale-up, dyeing

## 1 INTRODUCTION

Gold nanoparticles exhibit unique optical properties compared to bulk gold. These properties allow for potential applications in sensing and the dyeing of wool fibres. The latter exploits the intense surface plasmon resonances of gold nanoparticles, which absorb photons in the visible region of light. A novel proprietary approach for the synthesis of gold nanoparticle-wool fibre composites has been developed by Professor James H Johnston and Dr Kerstin A Lucas from Victoria University of Wellington and Noble Bond Ltd. [1-2]. Gold is associated with wealth and desire, and as such, gold nanoparticle-wool fibre composites are being fabricated into luxury garments, apparel, textiles and carpets for international markets.

These novel proprietary nanocomposites which are being produced and marketed by Noble Bond Ltd, have received extensive interest from international high end apparel and textile companies, who desire demonstration products for market appraisal. This necessarily required scaling-up the synthesis of gold nanoparticle-wool fibre composites from the laboratory-scale to the kilogram-scale.

The synthesis of gold nanoparticle-wool fibre composites has been optimised at the laboratory-scale by

Johnston and Lucas [1-3]. Examples of composites synthesised on the laboratory-scale are shown in Figure 1 below [3].

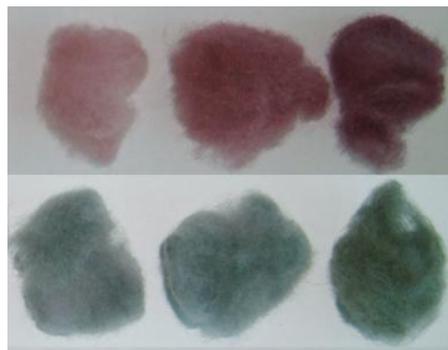


Figure 1: Gold nanoparticle-wool fibre composites using 0.1 g of top wool [3].

Gold nanoparticle synthesis is easily reproducible on the laboratory-scale and numerous methods are readily available. However, significant issues arise when a 100 L or larger volume of gold nanoparticles is synthesised, as uniform heating and mixing is more difficult to achieve on this scale. Additionally, the possibility of gold plating onto reaction vessel surfaces has to be obviated. Despite these difficulties, 100 L gold colloids have been successfully synthesised in this research.

Gold colloids and gold nanoparticle-wool fibre composites synthesised on the laboratory-scale and large-scale are synthesised and characterised in this paper. These results show that such nanomaterials can be successfully made on a larger than laboratory scale and provide insights into the scaling-up of other nanomaterials.

## 2 MATERIALS AND METHODS

### 2.1 Materials and Reagents

Wool fibres used in this research are shorn from New Zealand merino or crossbred sheep, and are provided through Professor James H Johnston's commercial industry partners.

The  $\text{HAuCl}_4$  solution that is used for gold nanoparticle synthesis was prepared by dissolving metallic gold in

concentrated aqua regia. The concentration of  $\text{HAuCl}_4$  in the solution is measured with atomic absorption spectroscopy.

All other chemicals were purchased from Sigma Aldrich.

## 2.2 Analysis Methods

UV-visible absorption spectroscopy was measured with an Agilent Diode Array Spectrophotometer. Transmission electron microscope (TEM) images of gold colloids were acquired with a JEOL 2010 High Resolution TEM instrument at 200 kV. Gold colloids were evaporated onto carbon-coated copper grids and then air dried prior to TEM analysis. Scanning electron microscope (SEM) analysis was undertaken with a JEOL 6500 F field emission gun SEM. Composite fibres are mounted on aluminium stubs with double sided carbon tape, coated with a JEOL JEC-560 carbon coater, and dried under vacuum overnight to gain optimal imaging conditions. Energy dispersive X-ray spectroscopy (EDS) was measured under low vacuum mode at 15 kV.

## 2.3 Gold Nanoparticle Synthesis

Gold colloids were synthesised utilising tannic acid (TA) or trisodium citrate (TSC) as dual-reducing and stabilising agents, using procedures based upon well-established literature methods [4-5]. The colloids are termed TA-Au NPs or TSC-Au NPs, referring to the stabilising agent used in their syntheses.

# 3 RESULTS AND DISCUSSION

## 3.1 Laboratory-scale Chemistry

Gold colloids were synthesised on the laboratory-scale and characterised with UV-visible spectroscopy, transmission electron microscopy (TEM) and scanning electron microscopy (SEM). TEM images of typical solutions of TA-Au NPs and TSC-Au NPs are shown in Figure 2, together with their UV-visible absorption profiles. The colour and hence UV-visible absorption spectrum of a gold colloid is indicative of the size, shape and polydispersity of gold nanoparticles in solution, and is a useful technique to compare between solutions. TEM images of TSC-Au NPs and TA-Au NPs are shown in Figure 3. The nanoparticles are spherical and approximately 10 nm in size.

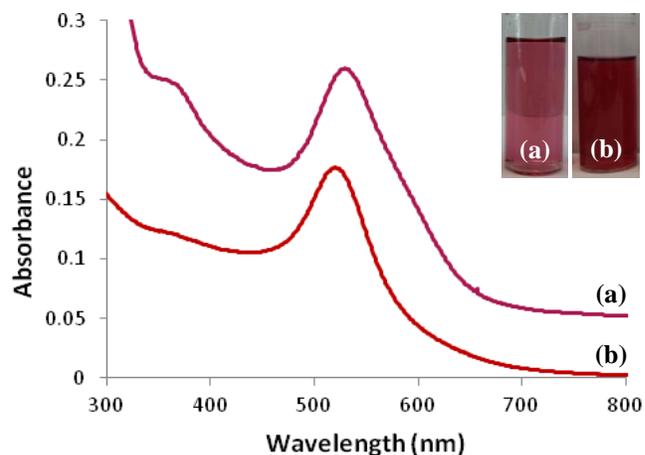


Figure 2: Solutions of TA-Au NPs (a) and TSC-Au NPs (b) synthesised on the laboratory-scale, and their UV-visible absorption spectra.

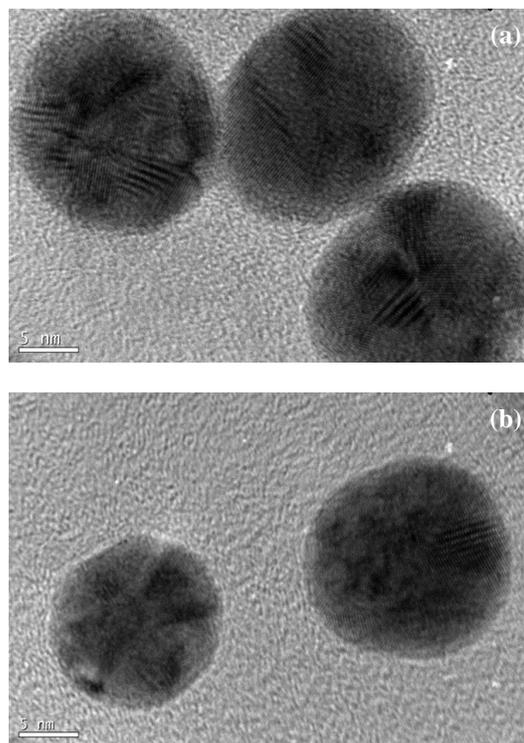


Figure 3: TEM images of TSC-Au NPs (a) and TA-Au NPs (b), where the scale bar reads 5 nm.

These gold colloids are used to dye wool fibres to produce novel hybrid nanocomposites using a proprietary method. A mauve coloured composite is shown in Figure 4 (inset). The SEM image in Figure 4 (a) illustrates the distribution of gold nanoparticles across an area of a wool fibre surface, where the gold nanoparticles appear as bright dots and clusters on the darker wool fibres. The gold content of the NPs is confirmed with Au EDS elemental analysis in Figure 4 (b), where areas of high concentration

of gold in the EDS map match the areas of high concentration of gold nanoparticles in the SEM image. The gold nanoparticles are bound to the wool fibres as shown by X-ray photoelectron spectroscopy studies [2].

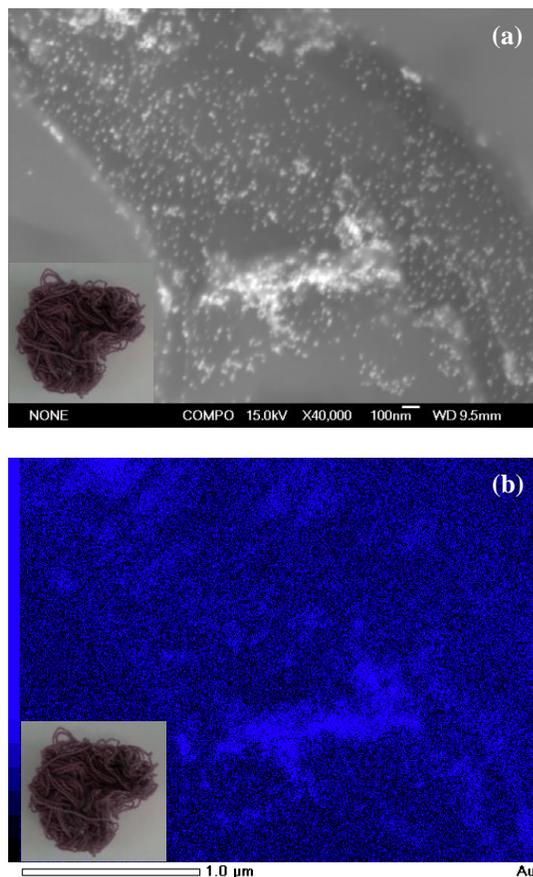


Figure 4: High magnification SEM image of a mauve coloured composite surface synthesised on the laboratory-scale in backscatter mode (a) and an elemental Au EDS map (b) of this area.

The even colouration of the composite fibres shown in Figure 4 (inset) is typical of those synthesised on the laboratory-scale, indicating uniform uptake and binding of gold nanoparticles by the wool fibres.

### 3.2 Scale-up Chemistry

The laboratory-scale synthesis of gold nanoparticle-wool fibre composites is easily reproducible and produces attractive, uniformly coloured composites. A number of difficulties are introduced when a 100 L or larger volume of gold colloid is synthesised, due to a variety of chemical processing conditions and unwanted gold deposition onto the surfaces of process equipment, as shown in Figure 5. The dyeing of wool fibres is likewise much more difficult on the large-scale, is completely different from conventional wool dyeing and specialist equipment is required.



Figure 5: Unwanted gold plating on a stainless steel reaction vessel.

TA-Au NPs and TSC-Au NPs have been synthesised using a stirred tank reactor to yield nanoparticles of a size and shape distribution that was comparable to those produced on the laboratory-scale (as evidenced by UV-visible spectroscopy and TEM analysis). The reactor used in these syntheses is shown in Figure 6 and a 100 L solution of TA-Au NPs synthesised in this way is shown in Figure 7.

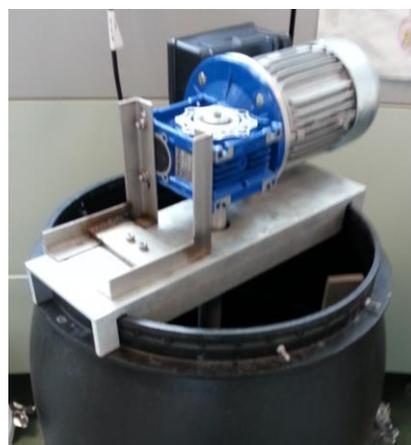


Figure 6: The barrel reactor that was designed for large-scale gold colloid synthesis with the mixer attached.



Figure 7: A 100 L solution of TA-Au NPs.

The UV-visible absorption spectra of the solutions of TA-Au NPs and TSC-Au NPs synthesised on the large-scale are comparable to those synthesised on the laboratory-scale, indicating a similar size and shape distribution of the particles that are formed. The above gold colloid was used to dye 2 kilogram quantities of merino yarn, to give the luxury composite fibres shown in Figure 8. Uniformly coloured yarn was produced, that was comparable to those dyed on the laboratory-scale.

These yarns have been successfully fabricated into consumer products, notably knitted apparel and bespoke carpets and rugs.



Figure 8: Large-scale mauve coloured gold nanoparticle-wool fibre composites synthesised in scale-up procedures.

## 4 CONCLUSIONS

Gold colloids were successfully synthesised on the laboratory-scale and large-scale, and used to produce gold nanoparticle-wool fibre composites with remarkable colour uniformity. These accomplishments have been important steps in the commercialisation of these proprietary nanocomposites, and are also applicable to the larger scale manufacturing of other nanoparticles and nanomaterials. This will aid in the transferral of novel nanoscience from the laboratory into the marketplace.

## 5 ACKNOWLEDGEMENTS

We gratefully acknowledge support from the MacDiarmid Institute of Advanced Materials and Nanotechnology and the Victoria University of Wellington Faculty of Sciences Strategic Research Grant, 2013.

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