Scale-up of Novel Gold Nanoparticle-Wool Fibre Composites: A Commercialisation Success Story

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ABSTRACT

Gold nanoparticles are known for their remarkable optical properties; they exhibit localised surface plasmon resonance bands in the visible region of the electromagnetic spectrum. This has led to their use as luxury dyes for the colouring of fine merino yarns. Gold nanoparticle-wool fibre composites are being fabricated into high-quality apparel for international markets. The innovative nanotechnology utilises the affinity of gold for sulfur-containing cystine residues in wool fibres to bind the gold nanoparticles. The uniform dyeing of wool fibres on the laboratory scale is easily reproducible. However, when the process is scaled-up, preventing colour variation across a kilogram of woollen yarn is a significant challenge. To overcome this, industrial package dyeing flow reactors were used to uniformly dye kilogram quantities of fine merino yarn. The pathway from synthesis in the laboratory to pilot-scale production of gold nanoparticle-wool fibre composites is presented.

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

1 INTRODUCTION

The development of novel nanocomposites of metal nanoparticles and natural or synthetic fibres has allowed the engineering of flexible materials with the desirable properties of metal nanoparticles. These multifunctional nanocomposites have numerous applications in many fields, including catalysis, sensing, medicine and textiles. The development of scalable and cost-effective methods to synthesise and uniformly disperse nanoparticles throughout a matrix has enabled the scale-up of such nanocomposites.

One type of novel nanocomposite uses gold nanoparticles as luxury dyes to colour merino wool fibres, by utilising the affinity of gold for sulfur-containing cystine residues in wool fibres. Gold nanoparticle-wool fibre composites were first developed by Professor James H Johnston and Dr Kerstin Lucas from Victoria University of Wellington and Noble Bond Ltd [1-3]. The innovative nanotechnology is being commercialised by Noble Bond Ltd. under the trade name Aulana™. These novel nanocomposites are used in the fabrication of high quality fashion apparel and luxury textiles for international markets.

The synthesis of gold nanoparticle-wool fibre composites has been optimised at the laboratory-scale by Johnston and Lucas. A range possible colours the nanocomposites is shown in Figure 1 below. The optical properties of gold nanoparticles are dependent on the size and shape of the particles. Thus, tuning the size and shape of the particles allows for a boutique colour range of nanocomposites, as evidenced in Figure 1, where the colour range includes pinks, purples and different shades of grey.

Figure 1: A range of colours of gold nanoparticle-wool fibre composites.

There are numerous size-controllable syntheses of gold colloids at the laboratory-scale that can uniformly colour wool fibres. However, when the process is scaled-up, nanoparticle synthesis is no longer trivial, and preventing colour variation across a sample is a significant challenge. We have reproducibly scaled up the synthesis of 100 L volumes of gold nanoparticles and used them to colour felted wool yarn used to fabricate luxury Aulana™ rugs [4].

The manufacture of luxury apparel requires fine merino yarns, the dyeing which typically utilises the package dyeing process, where dye liquor is pumped through a wool cone at relatively fast flowrates. This paper presents the journey from synthesis in the laboratory to pilot-scale production of gold nanoparticle-wool fibre composites in general terms only as the details are proprietary.

2 MATERIALS AND METHODS

2.1 Materials and Equipment

Finished yarns of New Zealand merino wool were provided through Professor James H Johnston's commercial industry partners.

The $HAuCl_4$ solution used in the synthesis of gold nanoparticles was prepared by dissolving metallic gold in concentrated aqua regia. Tannic acid and trisodium citrate were purchased from Sigma Aldrich.

Package dyeing pilot-scale equipment at Tararua Yarns Ltd in Levin, New Zealand, was used to colour merino yarn.

2.2 Gold Nanoparticle Synthesis

Solutions of gold nanoparticles were synthesised utilising tannic acid (TA) or trisodium citrate (TSC) as dual-reducing and stabilising agents, using procedures outlined in a previous publication [4].

2.3 Analysis Methods

UV-visible absorption spectroscopy was measured with an Agilent Diode Array Spectrophotometer. 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.

3 RESULTS AND DISCUSSION

Large volumes of gold colloids were synthesised in the stirred tank reactor shown below to yield nanoparticles of a size and shape distribution that was comparable to those produced on the laboratory-scale. Solutions of up to 100 L of gold nanoparticles were synthesised at a time.

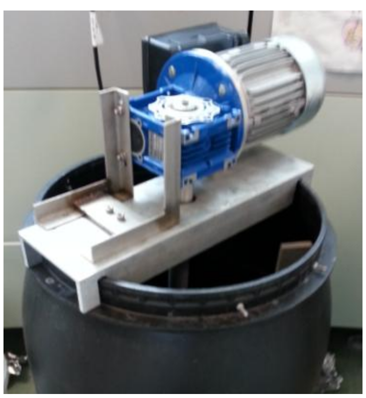

Figure 2: The barrel reactor that was designed for large-scale gold colloid synthesis with the mixer attached.

Images and UV-visible absorption spectra of the gold colloids synthesised in the barrel reactor and used for subsequent dyeing of fine merino yarns are presented in Figure 3.

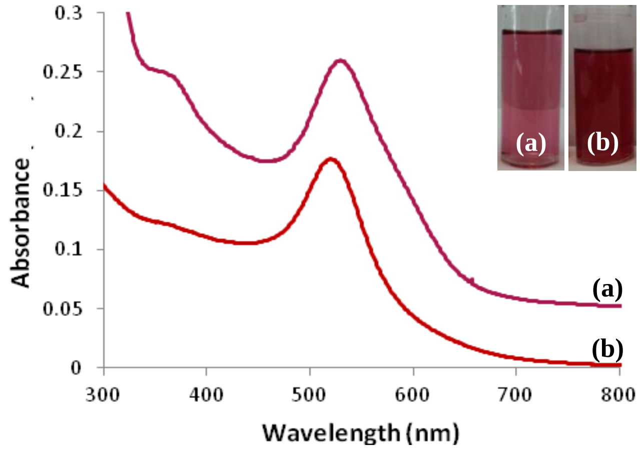

Figure 3: Images and UV-visible absorption spectra of solutions of gold nanoparticles synthesised with TA (a) and TSC (b).

The above gold colloids were used to dye merino wool yarn. Figure 4 presents the results of the synthesis of two mauve-grey composite samples that were synthesised under different reaction conditions. The image shows yarn taken from the inside, middle and outside of the dyepack, which is then knitted together to illustrate the colour uniformity throughout the dyepack. The composites in Figure 4 (a) exhibit a significant colour gradient throughout the sample, where gold nanoparticles were preferentially absorbed at the inside of the dyepack.

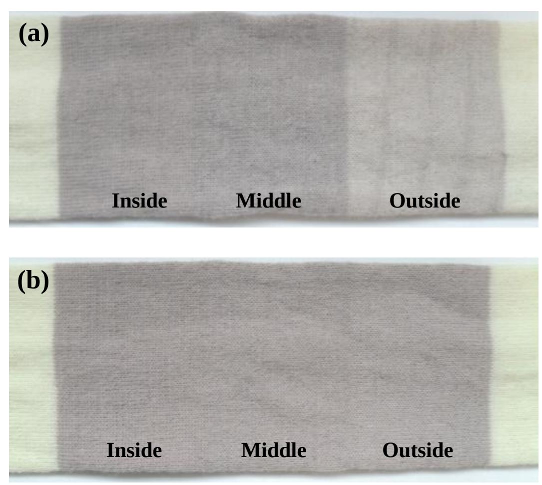

Figure 4: A representation of different areas of the dye pack of gold nanoparticle-wool fibre composites that were synthesised under different reaction conditions

The composites depicted in Figure 4 (b) exhibited much improved colour uniformity, with minimal variation of colour seen throughout the dye pack. This was achieved by altering the reaction conditions of the synthesis. The process was further improved to produce the uniformly purple coloured merino yarn shown in Figure 5, where there is no variation in colour across the sample.

Figure 5: A package dyed sample of gold nanoparticle-wool fibre composite fibres with uniform colouration.

SEM images of the surface of a composite merino fibres in Figure 5 are shown in Figure 6 below. The backscatter image at low magnification shows the presence of gold nanoparticles across the surface of the wool fibre, which appear as bright dots on the darker wool surface. Further magnifaction of the square area in Figure 6 (a) shows that the gold particles are in the nano-dimensions, and are typically monodisperse, with a few agglomerates of gold particles present. SEM images of composite fibres from different areas in the dye pack show a similar distribution of gold nanoparticles.

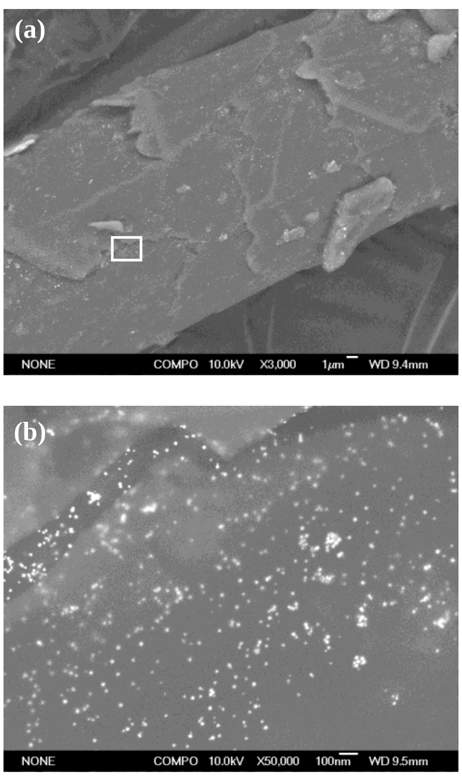

Figure 6: SEM micrographs in backscatter mode of the surface of the composite in Figure 5, where (b) is a magnification of the square area in (a).

4 CONCLUSIONS

The reproducible uniform colouring of fine merino yarn is a significant accomplishment of this work and has been an important step in the commercialisation of Aulana. This, together with the scale-up of the dyeing of felted carpet yarns [4], has allowed the reproducible supply of two distinct gold nanoparticle-wool fibre composite types for fine apparel and luxury rug fabrication respectively. The results of this work will assist the scaling-up of other novel nanocomposites that require large-scale metal nanoparticle synthesis or uniform absorption of metal nanoparticles by a fibrous matrix.

5 ACKNOWLEDGEMENTS

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