



# Long-Chain Hydrosilanes Mediated Phase Transfer of Aqueous Metal Nanoparticles

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**Abstract:** This research presents a new approach to the dispersion and stabilization of gold nanoparticles from aqueous to organic solution through the use of alkylsilanes. The unique phase activity of poly(hydro)silanes inspired the investigation of monomeric hydrosilanes as transfer agents. This method utilizes n-butylsilane, hexylsilane, octylsilane, and octadecylsilane as transfer agents to complete a ligand exchange, allowing the nanoparticles to flow into organic solution. These reactions were monitored and characterized through UV-Vis Spectrometry (UV-Vis), Fourier Transform Infrared Spectroscopy (FTIR), and Transmission Electron Microscopy (TEM). The gold and silver nanoparticles transferred using this protocol maintained their size and shape throughout the reaction. This phase-transfer reaction successfully transferred 2-AST stabilized gold and silver nanoparticles to a variety of organic solvents which remained stable for prolonged periods.

**Keywords:** hydrosilanes; nanoparticles; Organosols; dispersibility; octadecylsilane; octylsilane; hexylsilane; n-Butylsilane; n-(2-Aminoethyl)-3-aminosilanetriol; gold nanoparticles; phase transfer; hydrophobic agents; stability

## 1. Introduction

Nanoscale materials have provided a multitude of advancements through their unique physical, electrical, and optical properties [1–3]. The nanometer size of these materials endows them with properties that widely differ from their bulk and molecular counterparts [4]. Seminal discoveries and manipulation of the synthetic routes of nanomaterials have made it possible to generate particles in many different sizes and shapes such as spheres, rods, wires, planes, and more; depending on their intended use [5–7]. The unique properties observed at nanoscale can be applied to a new range of applications. For instance, with their high surface-area-to-volume ratio, nanoparticles can help to load a greater number of therapeutic ligands or labels for targeted delivery [8]. Another benefit of this property is that when nanoparticles are used as catalysts, the rate and reactivity are increased [4]. Thus, the unique properties of materials at nanoscale increases their effectiveness in various applications such as in catalysts, microelectronics, and drug-delivery systems, to name a few [9–11].

There have been multiple published protocols reporting the detailed synthesis of metallic nanoparticles in both polar and nonpolar solvents [7,12,13]. Each method has its advantages and disadvantages, depending on the intended end use and application. In typical procedures, nanoparticle synthesis is carried out in organic solvents through the extraction of metal ions from an aqueous phase, followed by reduction in the presence of a capping agent [14]. Some methods yield good control over size and monodispersity, but little control over the shape of the nanoparticles [14–16]. Post-synthetic modification of the nanoparticles is often a required step to tailor the chemical, physical, and optical properties for specific applications [17,18].



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**Copyright:** © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). It is desirable to develop methods and protocols which will open avenues for the transfer of metal nanoparticles between aqueous and organic media [17,19,20]. Novel metal nanoparticles have been commonly produced in aqueous solutions, as the synthesis involves a simple pathway [7]. Generally, they are transferred to organic media for more practical use through a variety of methods [14–17,20–24]. The phase transfer of metal nanoparticles is commonly carried out by ligand exchange, utilizing alkyl amines and thiols with differing chain lengths [7,14,16–25]. However, these methods are not compatible with certain types and sizes of nanoparticles; many require a prestabilization step [7,14,16–25]. Due to the variety of applications that require metallic nanoparticles in an organic solution, new transfer protocols to produce stable metallo-organic sols can be very useful and important.

Recent research in our group has shown that long-chain monomeric hydrosilanes can provide a new avenue to create particles with good stability while maintaining their reactivity [13]. In these studies, an interesting effect was observed during the storage of octadecylsilane (ODS)-stabilized silver nanoparticles [13]. After 24 h of storage, these particles settle at the bottom of the flask and upon centrifugation can be isolated and redispersed in other organic solvents [13]. Utilizing a stabilizing agent that has the ability to transfer the nanoparticles between phases would allow for their practical use in any number of applications [17,26–28]. This fascinating property led us to investigate ODS and other long-chain hydrosilanes as a potential phase transfer agent. In this paper, we present results of our investigations, which have led to uncovering of a new approach to the dispersion and stabilization of gold nanoparticles from aqueous to organic solutions using common alkylsilanes (Figure 1).



Figure 1. A schematic representation of the phase transfer process.

#### 2. Results

An aqueous solution of gold nanoparticles was prepared using a methodology developed in our laboratory [12]. This method involves the utilization of n-(2-Aminoethyl)-3-aminosilanetriol (2-AST) as both the reducing and stabilizing agent at 60 °C [12] (See Section 3). These nanoparticles were used as prepared for the phase transfer process. The exploratory studies of phase transfer of gold nanoparticles mediated by alkyl silanes were carried out using n-butylsilane (Scheme 1). The first indication of successful phase transfer was the change in the color of the organic layer. In the initial experiments, aqueous nanoparticles were vigorously stirred at 350–400 rpm with silane-containing toluene at room temperature. After 2 days, when the stirring was stopped, the toluene layer appeared purple/red, indicating the transfer of nanoparticles from the water layer to the toluene layer (Figure 2).



**Scheme 1.** Representation of the phase transfer of 2-AST gold nanoparticles through a ligand exchange with n-butylsilane.



**Figure 2.** Images of the reaction mixture, (**A**) before reaction has started and (**B**) after completion of the phase transfer with n-butylsilane.

Since gold nanoparticles are known to possess a surface plasmon band between 500–600 nm (depending on size and morphology), UV-Visible spectroscopy was used to effectively establish the presence of gold nanoparticles in the organic layer [1,29]. Figure 3 shows the progression of the phase transfer with n-butylsilane where an absorbance at 529 nm is used as an indicator of the transfer of nanoparticles from one phase to another. In addition, UV-Vis spectroscopy was also used to analyze the nanoparticle content of both the organic and aqueous layers.

After 1 h of reaction, a small peak at ~530 nm is obtained, indicating that some nanoparticles have been transferred to the organic layer. After 24 h, the nanoparticle content present in the toluene layer saw an increase while that of the aqueous layer decreased. After 2 days, it was observed that more than 30% of the nanoparticle concentration was transferred to organic layer. It was observed that prolonging the reaction time further, as well as increasing the temperature, did not improve the transfer to the organic layer. It is important to note that slower stirring speeds resulted in prolonged transfer times, as there was less surface contact created between the phases.

Fourier Transform Infrared spectroscopy (FTIR) is a very useful tool in identifying successful ligand exchange reaction. The Si-H bond on a silane can be identified by a strong band at 2280–2080 cm<sup>-1</sup>, which normally sees no interference from other bands [30]. Figure 4 illustrates the disappearance of the Si-H peak from the precursor n-butylsilane and appearance of a new peak centered at 2156 cm<sup>-1</sup> once the ligand exchange has been completed. The appearance of this new broad Si-H peak, from the remaining Si-H bonds, indicates the formation of a bond between the silane and the nanoparticle surface. This confirms that silanes can be effective in bonding and stabilizing the gold nanoparticles.



**Figure 3.** UV-Vis analysis of the nanoparticle-containing toluene layer of the phase transfer reaction mixture at different time intervals. n-Butylsilane was the phase transfer agent.



**Figure 4.** Infrared Spectrum Analysis of (**A**) n-butylsilane vs. (**B**) dried Au nanoparticles after phase transfer with n-butylsilane.

The ligand transfer is also verified by comparing the IR spectra of 2-AST stabilized gold nanoparticles and the IR spectra of phase-transferred silane stabilized gold nanoparticles. As is evident from the spectra Figure 5A, specific peaks associated with 2-AST have disappeared. This IR study clearly shows the differentiation in capping agents in aqueous and organic phases.

Transmission Electron Microscope (TEM) imaging was also used to monitor the progression of the reaction as more nanoparticles move into the organic phase. The micrographs show the gold nanoparticles, surrounded by the silane, dispersed in the organic medium. As the reaction progresses, a greater number of nanoparticles can be visualized in the organic phase. TEM analysis of the toluene layer was performed, as shown in Figure 6, after completion of the reaction. The nanoparticles can be seen dispersed in a "bubble" of the silane throughout the organic phase.



**Figure 5.** Infrared Spectrum Analysis of (**A**) dried 2-AST gold Nanoparticles vs. (**B**) dried Au Nanoparticles after phase transfer with n-butylsilane.

The silane has the ability to bond to the nanoparticles, allowing them to move into an organic solvent. Some nanoparticles remain in the aqueous layer after transfer, as seen in Figure 7. Particle size analysis was not done for the particles which are remaining in aqueous layer (Figure 7). After investigation and optimization of n-butylsilane as a phase transfer agent, other alkylsilanes were explored to determine if modification of the chain length affects the rate or results of the reaction (Figures 8–10). A comparison of the effectiveness of the transfer under identical conditions is summarized by UV-Vis spectroscopy in Figure 11.



**Figure 6.** TEM micrographs of gold nanoparticles transferred with n-butylsilane in toluene. ((**A**) 30 k zoom (**B**) 60 k zoom).



**Figure 7.** TEM micrographs of the aqueous layer after completion of the reaction. ((**A**) 10.0 k zoom. (**B**) 60.0 k zoom).



**Figure 8.** TEM micrographs of gold nanoparticles in toluene with octylsilane as the phase-transfer molecule. ((**A**) 5.0 k zoom. (**B**) 30.0 k zoom).



**Figure 9.** TEM micrographs of gold nanoparticles in toluene with hexylsilane as the phase-transfer molecule. ((A) 15.0 k zoom. (B) 50.0 k zoom).



**Figure 10.** TEM micrographs of gold nanoparticles in toluene with octadecylsilane as the phase-transfer molecule. ((A) 30 k zoom. (B) 60 k zoom).



**Figure 11.** UV—Vis analysis of the nanoparticle—containing toluene layer of the phase transfer reaction with varying silane chain length.

All the primary alkylsilanes which were investigated had the ability to transfer roughly 35% of the nanoparticles from aqueous media to organic media. Particle size analysis, carried out using Image J, showed that there was no substantial change in particle size after the transfer. The change in the particle size was found to be between 0.6 to 1.5 nanometer range. In Table 1, a comparison of pre- and post-transfer nanoparticle sizes is summarized for each silane phase transfer agent. In all the cases, gold nanoparticles transferred with alkylsilanes into organic solutions were found to be stable for prolonged time periods. UV-Vis spectra of the transferred particles shows their size is dependent on the silane's chain length. The arrangement of the longer chain silanes on the surface of the nanoparticle will pack in a different fashion than the shorter chain silanes due to steric effects as they assemble on the nanoparticle surface [31]. The packing of the stabilizing silanes can be random, and some longer alkyl chains may be bent, causing steric interactions which prolong the reaction rate and quick adsorption of the transfer agent [31]. It seems plausible that the relatively short alkyl chains on n-butylsilane do not stabilize the particles as well as the longer chained silanes, and therefore show a slight increase in size. UV-Vis spectra shows the localized surface plasmon resonance (LSPR) shifts only slightly upon transfer, which suggests that particle aggregation and size change is negligible [20]. The

bathochromic shift of the transferred particles is also enhanced by the higher refractive index of toluene compared to water [20].

Silanes	Nanoparticle Size Post-Transfer	Original Nanoparticle Size	Change in Size	Standard Deviation
Octadecylsilane (ODS)	23.7 nm	22.6 nm	+1.1 nm	2.965 nm
Octylsilane	23.2 nm	22.6 nm	+0.6 nm	3.419 nm
Hexylsilane	23.4 nm	22.6 nm	+0.8 nm	3.381 nm
Butylsilane	24.1 nm	22.6 nm	+1.5 nm	3.152 nm

Table 1. Comparative study of alkylsilanes as phase transfer agents.

<sup>1</sup>H-NMR spectroscopy was used to confirm the composition of the silane-protected nanoparticles. <sup>1</sup>H-NMR studies confirmed that n-butylsilane couples to the nanoparticle surface through the breakage of Si-H bonds and formation of Si-Particle bonds. A schematic representation of the phase-transfer process with alkylsilanes is depicted in Scheme 2. The spectra of n-butylsilane shows a triplet at 3.52 ppm, corresponding to the three Si-H protons. <sup>1</sup>H-NMR of the transferred n-butylsilane nanoparticles exhibit a broad peak, shifted downfield to 5.02 ppm. This shift indicates that the Si-H bonding environment has dramatically changed due to their interaction with the metal nanoparticle surface. <sup>1</sup>H NMR spectra can be found in the Supplementary Information.



Alkylsilane Stabilized Nanoparticles in Toluene

**Scheme 2.** A schematic representation of the phase transfer of 2-AST stabilized gold nanoparticles using short and long chain alkylsilanes. Scheme adapted from Chauhan et al. [12].

In order to examine the wider applicability of this method, silver nanoparticle phase transfer was also conducted (Scheme 3). The 2-AST stabilized silver nanoparticles were synthesized according to previously published protocol and phase transfer was attempted using the alkylsilanes [12]. n-Butylsilane was able to successfully transfer aqueous silver nanoparticles with little-to-no change in morphology and dispersity (Figure 12).



**Scheme 3.** A schematic representation of the phase transfer of 2-AST stabilized silver nanoparticles using short- and long-chain alkylsilanes. Scheme adapted from Chauhan et al. [12].



**Figure 12.** TEM micrographs of 2-AST silver nanoparticles in toluene with n-butylsilane as phase transfer molecule. ((**A**) 40.0 k zoom. (**B**) 100.0 k zoom).

Known phase-transfer agents were also used to conduct a comparative study of the effectiveness of alkylsilanes as transfer agents for 2-AST stabilized nanoparticles. Known transfer agents such as tetra-butyl ammonium hydrogen sulfate (TBAHS) and Hexade-cylamine (HDA) were investigated [25,32]. TBAHS was unsuccessful in transferring the aqueous 2-AST gold nanoparticles as the reaction lead to the decomposition of the particles. HDA was not an effective phase transfer molecule for 2-AST nanoparticles, as after 7 days of the reaction; almost no particles had been transferred. The use of 2-AST as a stabilizing agent seems to be required for successful transfer of nanoparticles with alkylsilanes.

To test this hypothesis, citrate gold nanoparticles were synthesized by the Turkevich method and their transfer with n-butylsilane was attempted [33]. Known transfer agents, TBAHS and HDA, were also tested for transfer effectiveness. The citrate-stabilized gold nanoparticles did not undergo phase transfer using n-butylsilane or TBAHS as the transfer molecule. However, HDA was successful in facilitating transfer to toluene. As silanes are prone to oxidation in the presence of metal complexes, the citrate particles may be oxidizing the silane, thereby reducing its activity for phase transfer. This supports the hypothesis

that the effectiveness of alkylsilanes as phase transfer molecules is dependent on 2-AST as the stabilizing agent.

#### 3. Materials and Methods

**Materials:** All reagents were used from manufacturer with no further purification. Reactions were monitored for up to 3 days via UV-Vis spectroscopy and TEM to monitor the rate of transfer. All the reactions were carried out under a vent hood with the proper safety equipment. 2-AST Gold nanoparticles were synthesized using n-(2-aminoethyl)-3aminosilanetriol (2-AST) as both a reducing and stabilizing agent [12]. Gold (III) Chloride Trihydrate was used as the gold precursor. Alkylsilanes utilized as phase transfer molecules include octadecylsilane, octylsilane, hexylsilane, and n-butylsilane. Phase transfer was attempted using a variety of organic solvents such as toluene, hexane, and benzene.

**Synthesis of 2-AST Stabilized Gold Nanoparticles:** Gold nanoparticles were synthesized according to the procedure adopted from Chauhan et al., 2016 [12]. In this synthesis, 0.0357 g of gold (III) chloride trihydrate was placed directly into a 50 mL Erlenmeyer flask. A total of 20 mL of water and a magnetic stirrer bar were added to the flask. The flask was then placed in an oil bath situated to maintain 60 °C throughout the reaction. 0.3 mmol of 2-AST was slowly added to the reaction via a micropipette. The reaction was then monitored using UV-Vis Spectroscopy readings between 250–800 nm at half-hour intervals. The reaction mixture showed a peak at 529 nm after 30 min of reaction.

**Phase Transfer Protocol:** 

- (a) Phase transfer with n-butylsilane: A ligand exchange was carried out on 2-AST gold nanoparticles according to the following procedure: 3 mL of 2-AST gold nanoparticles, as synthesized, were added to a 25 mL Erlenmeyer flask; 5 mL of toluene and a magnetic stirrer bar were then added to the flask; to this solution, 1 mmol (130  $\mu$ L) of n-butylsilane was micropippetted into the reaction flask with vigorous stirring. After 1 h of reaction, the toluene layer showed a slight absorbance centered at 538 nm, indicating the start of the transfer of nanoparticles into toluene layer. The reaction mixture was monitored for 2 days periodically using UV-Vis Spectroscopy. The maximum particle transfer was achieved during this time. No increase in the absorbance was observed after 2 days.
- (b) Phase transfer with hexylsilane: A ligand exchange was carried out on 2-AST gold nanoparticles according to the following procedure: 3 mL of 2-AST gold nanoparticles, as synthesized, were added to a 25 mL Erlenmeyer flask: 5 mL of toluene and a magnetic stirrer bar were then added to the flask: to this solution, 1 mmol (161.9  $\mu$ L) of hexylsilane was micropippetted into the reaction flask with vigorous stirring. After 1 h of reaction, the toluene layer showed a slight absorbance centered at 532 nm, indicating the start of the transfer of nanoparticles into toluene layer. The reaction mixture was monitored for 2 days periodically using UV-Vis Spectroscopy. The maximum particle transfer was achieved during this time. No increase in the absorbance was observed after 2 days.
- (c) Phase transfer with octylsilane: A ligand exchange was carried out on 2-AST gold nanoparticles according to the following procedure: 3 mL of 2-AST gold nanoparticles, as synthesized, were added to a 25 mL Erlenmeyer flask; 5 mL of toluene and a magnetic stirrer bar were then added to the flask; to this solution, 1 mmol (193.5  $\mu$ L) of octylsilane was micropippetted into the reaction flask with vigorous stirring. After 1 h of reaction, the toluene layer showed a slight absorbance centered at 528 nm, indicating the start of the transfer of nanoparticles into toluene layer. The reaction mixture was monitored for 3 days periodically using UV-Vis Spectroscopy. The maximum particle transfer was achieved during this time. No increase in the absorbance was observed after 3 days.
- (d) Phase transfer with octadecylsilane: A ligand exchange was carried out on 2-AST gold nanoparticles according to the following procedure: 3 mL of 2-AST gold nanoparticles, as synthesized, were added to a 25 mL Erlenmeyer flask; 5 mL of toluene and

a magnetic stirrer bar were then added to the flask; to this solution, 1 mmol of ODS was introduced into the reaction flask with vigorous stirring. After 1 h of reaction, the toluene layer showed a slight absorbance centered at 530 nm, indicating the start of the transfer of nanoparticles into toluene layer. The reaction mixture was monitored for 3 days periodically using UV-Vis Spectroscopy. The maximum particle transfer was achieved during this time. No increase in the absorbance was observed after 3 days.

(e) Characterization of Gold Nanoparticles: UV-Visible absorption spectra, along with Transmission Electron Microscopy (TEM) micrographs were used for periodic analysis of the nanoparticles. The <sup>1</sup>H-NMR studies were used to analyze the incorporation of stabilizing ligands onto the nanoparticles. (See Supplementary Information) Fourier Transform Infrared Spectroscopy was used to analyze both layers of the solution after drying. The <sup>1</sup>H-NMR spectral peaks of n-butylsilane and n-butylsilane stabilized nanoparticles are given below. n-Butylsilane:  $\delta$ H (400 MHz; CDCl<sub>3</sub>;) 0.79, 0.93, 1.43, 2.20, 3.52, 4.65. n-Butylsilane Gold Nanoparticles:  $\delta$ H (400 MHz; CDCl<sub>3</sub>; Toluene) 0.41, 0.96, 1.20, 1.68, 2.33, 2.60, 5.02, 7.28, 7.44, 7.51.

## 4. Conclusions

The phase transfer of gold nanoparticles from aqueous to organic solution was achieved through the use of alkylsilanes, causing a ligand exchange with the nanoparticles, allowing for their transition into the organic medium. Through optimization of the reaction conditions and varying alkylsilane chain length, it was found that all the tested silanes could facilitate phase transfer to organic media. However, it appears that the phase transfer of the nanoparticles with alkylsilanes is dependent on 2-AST as the precursor stabilizing agent. Without the use of 2-AST particles, phase transfer will not be supported. The aqueous layer post-transfer is left with a 2-AST gel, with some of the original particles trapped inside. This method allows for the transfer of gold nanoparticles while simultaneously forming a gold-nanoparticle-impregnated 2-AST gel. Such types of metal-impregnated gels can open up new possibilities to give rise to the synthesis of novel nanocomposites. Exploration of functional nanocomposites fabrication is a very active area of research and is very useful for future applications. We are actively exploring such synthetic strategies and will be presenting the results in due course [34]. The timescale for the phase transfer of gold nanoparticles decreased with decreasing alkyl chain length of the silane. This proposed method for the phase transfer has been effective in transferring aqueous gold nanoparticles to a number of organic solvents such as Toluene, Benzene, and Hexane. Future endeavors may include investigation into the effect of pH and nanoparticle shape on the efficiency of phase transfer. Phase transfer of platinum and palladium 2-AST nanoparticles will also be tested.

**Supplementary Materials:** The following supporting information can be downloaded at: https: //www.mdpi.com/article/10.3390/macromol2020009/s1, FTIR spectra for 2-AST, 2-AST stabilized nanoparticles, aqueous 2-AST gel after transfer, n-butylsilane, n-butylsilane stabilized nanoparticles, octadecylsilane (ODS) stabilized nanoparticles. 1HNMR Spectra of Figure S1: n-butylsilane and Figure S2: n-butylsilane stabilized nanoparticles in toluene. TEM Images of 2-AST nanoparticles, n-butylsilane nanoparticles, hexylsilane, octylsilane, and octadecylsilane (ODS).

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