

Research Article

Parameters Affecting the Microwave-Assisted Polyol Synthesis of Silver Nanorods

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The effects of salt chemistry and concentration on the morphology and yield of silver nanorods produced by the microwave-assisted polyol synthesis are reported. Compared to NaCl, iron and copper salts reduced nanorod yield and length and increased diameter. This is in stark contrast to expectations based on the slower traditional polyol process. The role of the cation was further explored using MgCl₂ and MnCl₂ which showed different concentration dependent effects on nanorod diameter. In addition, the morphology and yield of nanomaterials was found to be significantly influenced by small variations in the procedure including container shape and the time between reactant mixing and microwave heating. These results highlight that, while the microwave-assisted process is very promising, results cannot be directly anticipated based on the traditional process, and the synthesis is very sensitive to procedural changes.

1. Introduction

In the last ten years, there has been significant interest in discovering scaleable routes for producing inorganic nanorods and nanowires. Common routes include the use of supercritical CO₂, synthesis in surfactant templated systems [1, 2], and directed growth methods such as the polyol process. While many methods have been published in the literature, reproducibility, particularly with regard to shape, size, and yield has been an ongoing challenge [3]. For example, the seed-mediated, surfactant-assisted synthesis of gold [4] was first reported in 2001. However, researchers were initially frustrated in reproducing results. It took over nine years to understand the effects of seed aging time [5], method of mixing the seed and growth solutions [5], variations in salt concentration, temperature, growth time [6, 7], and even trace impurities of iodide in the cetyltrimethylammonium bromide (CTAB) surfactant [3, 8] on the yield, shape, and size of the resulting nanoparticles.

Similarly, the microwave-assisted polyol process has shown promise for producing a broad range of spherical nanoparticles, nanowires, and other shapes. However, there has been little exploration on how changes in reactants and pro-

cedure affects shape and yield. There is ongoing interest in silver nanomaterials due to silver's antimicrobial properties. In addition, the size-dependent surface plasmon resonance (SPR) and local electric field strength combined with chemical sensitivity make silver attractive for chemical sensing applications based on surface-enhanced Raman-scattering (SERS) [9, 10]. More recently, silver nanorods and nanowires have also garnered significant interest for electronics. Silver's high electrical conductivity (63.0×10^6 S/m for Ag versus 60.7×10^6 S/m for Cu) makes silver nanowires interesting for interconnects [11]. This high conductivity coupled with the transparency that can be achieved at small-size scales also makes nanosilver based-materials promising candidates for the flexible electrodes needed in advanced displays, touch screens, and the next generation of solar cells [12]. Another application where one-dimensional silver nanostructures are beneficial is in the development of thermally and electrically conductive composites. Currently these materials utilize silver flake [13]; replacing the flake with silver nanowires could significantly reduce the loading required to achieve geometric percolation and reduce the cost of these materials. In these and other applications, size and shape of the nanomaterials are critical parameters that determine

performance. The percolation threshold of composite materials is directly related to the size and shape of the filler; smaller-diameter longer rods have a much lower percolation threshold than shorter larger-diameter rods. Transparency and flexibility of composite electrodes are dependent on the aspect ratio of the nanorods utilized. To commercially realize the potential applications of silver nanorods, it will be necessary to synthesize them at reasonable yields with control over size and shape. Ideally, the synthesis should be simple to perform and should not require the use of exotic reactants or conditions.

Several approaches have been explored for the production of silver nanowires including the use of nanoporous templates [14], hydrothermal synthesis [15], DNA templating [16], and wet chemical synthesis methods including the polyol process [4, 11, 17]. Most of these processes are complicated, and some do not produce products that are suitable for use in many applications. For example, the nanoporous templating process shows good control over the diameter but results in polycrystalline rods that strongly adhere to one another. As a result, dispersion of the nanoparticles in solution or within a polymer matrix is difficult. In contrast, the nanorods produced by the polyol process are easily separated from one another making them suitable for many applications. In the polyol process Ag^+ is reduced to Ag^0 by acetaldehyde which is generated by the thermal decomposition of a polyol, usually ethylene glycol, at the reaction temperature [18]. The reduced silver nucleates and grows by Oswald ripening and diffusion of newly reduced silver to the surface. Polyvinyl pyrrolidone (PVP) helps to stabilize the growing particles and directs growth in one direction by preferentially coordinating with one of the crystal faces limiting crystal growth in directions normal to that face. While the polyol process can be utilized to produce easily separable nanorods, the standard process requires careful control over reaction rates to tailor particle geometry. Several morphologies can be produced by controlling reaction conditions such as the concentration of Cl^- and H^+ , temperature, presence or absence of O_2 , and by controlling the reaction rate by dropwise addition of reactants [19, 20]. In addition, several “control” agents have been examined in the polyol process for their ability to control particle geometry. Fe(II) was reported by Wiley et al. to control the reaction through the oxidative etching of the silver seeds [19]. Chen et al. later demonstrated the reduction in silver nanowire diameter by the addition of $\text{Fe}(\text{NO}_3)_3$ [21]. This effect was explained through the process of etching the silver surface, the removal of oxygen from the surface, or the ability of metals with multiple valence states to affect the overall rate of reduction of Ag^+ . Other control agents that have been examined include salts of potassium, copper, and palladium and sodium sulfate [19, 21–23].

Recently there has been a significant interest in utilizing microwave heating to speed reaction rates and reduce the need for expensive catalyst [24]. Gou et al. demonstrated that a microwave-based polyol synthesis of silver nanorods can be completed in less than five minutes compared to several hours in the traditional process [25]. However, while several control agents have been explored in the traditional process, only NaCl and NaS have been examined in the microwave-

assisted process [25, 26]. The anions form AgCl salts or Ag_2S colloids and limit the amount of Ag^+ ions present in the solution that can be reduced to Ag^0 . This effectively controls the overall formation of Ag^0 , thus, allowing for directed growth to occur without the continued formation of new nuclei. In the case of Na_2S , a wide variety of shapes including rods and cubes have been synthesized. Several studies on the traditional polyol process have found that significant control over the rod morphology can be obtained by utilizing different metal chlorides, FeCl_3 , CuCl_2 , and KCl, for example. However, to date only sodium has been evaluated as the cation in the microwave-assisted process. This work extends understanding of how to control the synthesis of silver nanorods in the microwave-assisted polyol process. A range of salts were evaluated including NaCl, KCl, MgCl_2 , CaCl_2 , CuCl_2 , FeCl_3 , and MnCl_2 . The results indicate that some salts that are advantageous in the slower process are not suitable for the microwave-assisted process and that a previously unreported salt, MnCl_2 , is capable of producing small-diameter nanorods with a high aspect ratio.

2. Experimental

2.1. Materials and Equipment. Silver nitrate (AgNO_3) (>99%) was purchased from Sigma-Aldrich, Poly(vinyl pyrrolidone) (PVP) (M.W. = 58000) from Acros Organics, salts (NaCl, KCl, CaCl_2 , FeCl_3 , CuCl_2 , MnCl_2 , and MgCl_2) and ethylene glycol (EG) were purchased from Fisher-Scientific and used as received. Two microwaves were used in this work, a commercially available household microwave (General Electric model JES1855) and a 3200 W continuously variable power microwave from Microwave Research Labs (model BP 211). Three reaction vessel geometries were evaluated: a 5 cm diameter glass beaker, a 7 cm diameter glass beaker, and 10 cm diameter glass dish.

2.2. Methods. Figure 1 shows the schematic representation of the synthesis process used in this study. Three stock solutions were prepared separately: AgNO_3 in EG (0.026 M), PVP in EG (0.05 M), and salt solution (various concentrations). All stock solutions were prepared just prior to use and sonicated for 5 minutes to aid dissolution and ensure uniform dispersion of the solutes. After preparation of the three solutions, 20 mL of the AgNO_3 solution was placed in the reaction vessel followed immediately by 20 mL of the PVP solution and then by 20 mL of the salt solution; the total reaction volume was 60 mL. For all experiments, except investigations of container shape, the reaction vessel was a 300 mL glass container 5 cm in diameter. Immediately upon the addition of the salt solution, the entire dispersion became opaque with an opalescent color due to the formation of AgCl colloidal particles. The dispersion was mixed by swirling the container for 10–15 seconds followed by bubbling dry N_2 through the container at a flow rate of 50 cc/min for 1 min. The dispersion was then transferred to the microwave. When the household microwave was used, the power setting was at level 4 resulting in pulsed power of 13 seconds on and 17 seconds off. Tests with water indicated that the average delivered power under these conditions was 350 W. Total microwave

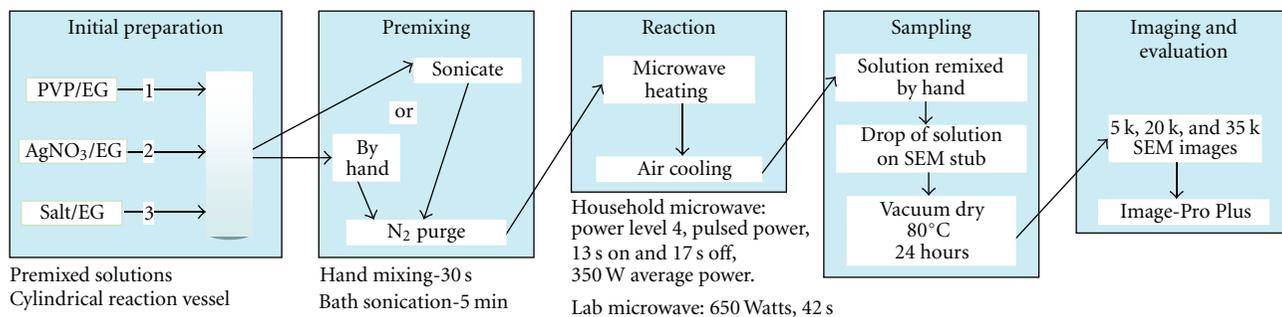


FIGURE 1: Schematic representation of synthesis, sample preparation, and evaluation processes used in this work.

time in this case was 2 minutes (four full cycles). The rotating turntable was off during the heating process. The laboratory microwave from Microwave Research Labs had continuous power control; when this oven was used, the power level was adjusted to 650 W and the reaction time was limited to 42 sec. Upon completion of the reaction, the dispersions were allowed to cool in air for 30 minutes. Samples were taken immediately after cooling for characterization.

2.3. Characterization. The reaction product was mixed by swirling the container for 30 seconds just prior to sampling with a disposable transfer pipet. Drops of the reaction product were placed on aluminium stubs coated with carbon tape and vacuum dried at 80°C for 24 hours to remove EG. The samples were imaged using a JEOL 7000 field-emission scanning electron microscope (SEM) without sputter coating. Since the coating on the nanorods, PVP, is not conductive, there is some hazing of the rod edges. High-magnification images (35 k magnification) were used to determine rod diameters. To measure length, lower-magnification images (5 k magnification) were used. The number of nanorods present in a 2 by 3 micron grid was determined in a 20 k magnification image and used as an indication of rod yield. All rods that intersected the measurement area were counted. At least three images were taken at each magnification, for each sample to account for sample variability across the stub. In some work, optical imaging was used to determine a rod/particle ratio. For this analysis, images were taken on Nikon Eclipse 80i at 60x magnification with 1.4 NA. These images were analyzed using Image Pro to determine the number of rods and particles in a fixed area.

3. Results and Discussion

3.1. Effect of Salt Concentration. Four salts, NaCl, KCl, MgCl₂, and CaCl₂, were tested to determine the effect of changing the cation within group I and II elements on the rod diameter, length, and nominal yield. Gou reported that the most favourable Cl⁻ to AgNO₃ ratio for the microwave-assisted synthesis was between 1 : 6 and 1 : 3 by mole when using NaCl as the salt [25]. Therefore, for NaCl and KCl, a Cl⁻ : AgNO₃ ratio of 1 : 6 was evaluated. For MgCl₂ and CaCl₂, both 1 : 6 and 1 : 3 ratios were evaluated. Figure 2 shows representative samples of the highest-magnification SEM images taken; these images, with others, were used to

evaluate the rod diameter. Table 1 provides the diameters, lengths, and rod counts for all salts evaluated in this study. Of the four salts evaluated from group I and II, MgCl₂ resulted in the smallest-diameter rods. The rod diameters were 38, 36, and 31 nm for NaCl, KCl, and MgCl₂, respectively. In contrast to the reduction in rod diameter achieved by changing the salt to MgCl₂, use of CaCl₂ resulted in larger diameter rods and a significant increase in the polydispersity of the rod diameters.

Both Wiley and Korte's work demonstrated that both the cation and the anion play a role in the formation and growth of silver nanowires [19, 23]. The anion, Cl⁻, is thought to act as an electrostatic stabilizer for the initially formed seeds and has also been postulated to limit the availability of Ag⁺ ions, thereby, controlling the overall reduction of Ag⁺ to Ag⁰ allowing directed growth to occur. These effects suggest that larger concentration would be more favourable. On the other hand, high concentration of Cl⁻ ion leads to etching of twinned seeds, suggesting an optimum concentration range for Cl⁻. In this study, at least two concentrations of salt were tested for those cations with a valance greater than one. For the divalent materials, tests were performed with the ratio of Cl⁻ to Ag at both 1 : 3 and 1 : 6; these are the limits suggested by Gou as the "best" for forming Ag nanorods in the microwave-assisted process [25]. In the case of Fe, the Cl⁻ to Ag ratios tested were 1 : 6 and 1 : 2. As shown in Table 1, in every case higher Cl⁻ concentrations resulted in fewer rods. In fact, at the higher of the two concentration for FeCl₃ evaluated, no rods were formed. The effect of concentration on the rod diameter and length was less clear. For group I and II elements at a Cl⁻ to AgNO₃ ratio of 1 : 6, the longest rods were obtained from KCl, and the highest number of rods was obtained from NaCl. Changing the Cl⁻ to Ag ratio to 1 : 3 for MgCl₂ and CaCl₂ affected the diameter, length, and rod number. Higher MgCl₂ concentrations resulted in significantly fewer, much shorter, and smaller diameter rods, aspect ratio (AR) ~46. A similar change with CaCl₂, however, resulted in fewer, slightly longer, smaller-diameter rods, AR ~237.

In the traditional polyol process, the presence of disassociated O₂ on the seed surface blocks the further deposition of Ag, limiting seed growth [19]. Korte et al. limited this effect by adding cations with multiple valence states, Fe⁺³ and Cu⁺², which are capable of scavenging oxygen from the growing silver surface resulting in faster growth and longer

TABLE 1: Rod diameter, length, and number of rods produced for salts evaluated in this study. ^aNominal aspect ratio is the mean measured length divided by the mean diameter. ^bThe number of rods intersecting a 2 by 3 micron grid, multiple images taken at various location on stub.

Salt	Atomic radius (metal) (pm)	Concentration (mM)			Ag rod properties (mean and Std. Dev.)			
		Metal	Chlorine	Cl: AgNO ₃ ratio	Diameter (nm)	Length (μm)	Nominal AR ^a	No. of rods ^b
NaCl	186	4.3	4.3	1:6	38.6 (9.1)	7.6 (1.1)	197	60 (9)
KCl	227	4.3	4.3	1:6	35.6 (9.0)	9.1 (1.3)	255	51 (4)
MgCl ₂	160	2.15	4.3	1:6	31.4 (4.5)	3.7 (1.2)	118	49 (9)
		4.3	8.6	1:3	26.1 (6.7)	1.2 (0.3)	46	12 (3)
CaCl ₂	197	2.15	4.3	1:6	41.0 (13.2)	7.4 (1.5)	180	58 (9)
		4.3	8.6	1:3	35.8 (8.8)	8.5 (1.4)	237	30 (3)
MnCl ₂	127	2.15	4.3	1:6	31.9 (8.6)	8.4 (1.4)	263	61 (6)
		4.3	8.6	1:3	41.8 (10.5)	7 (1.8)	167	29 (3)
FeCl ₃	126	1.43	4.3	1:6	57.3 (17.8)	0.5 (0.2)	9	23 (6)
		4.3	12.9	1:2	No rods found			
CuCl ₂	128	2.15	4.3	1:6	118.8 (52.7)	2.7 (1.3)	23	13 (2)
		4.3	8.6	1:3	168.7 (48.4)	1.3 (0.6)	8	9 (1)

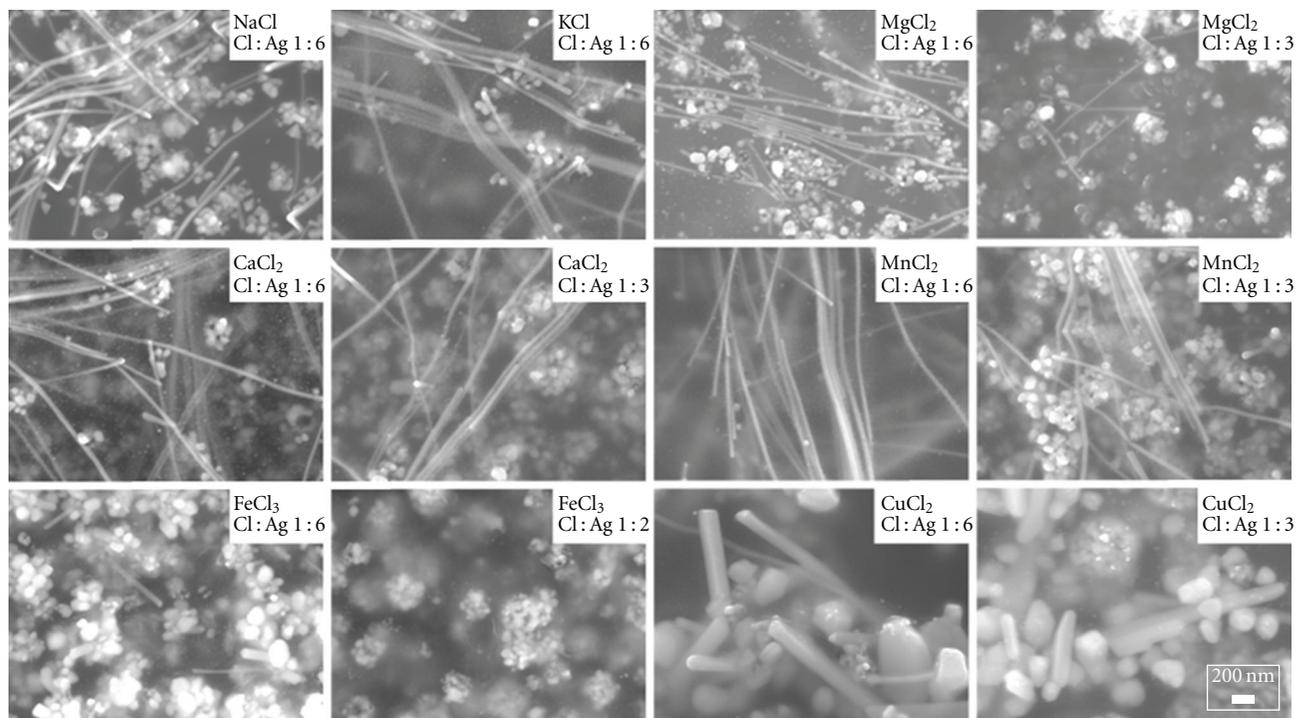


FIGURE 2: SEM images, magnification 35 k of silver nanomaterials showing the shapes, range of diameters, and lengths produced.

rods [23]. To determine if this effect exists when conducting microwave synthesis, chloride salts of the transition metals Mn, Fe, and Cu were evaluated in this work. Table 1 provides the measured diameter, length, and the normalized number of rods obtained when these salts were used in the microwave-assisted polyol process. In contrast to previous work on the traditional polyol process, an increase in rod

formation when using CuCl₂ and FeCl₃ versus the use of NaCl in the microwave-assisted process was not observed. The rods produced at all concentrations of FeCl₃ and CuCl₂ tested were fatter, shorter, and fewer in number. In previous studies, the reaction was conducted over a period of several hours, while in the current study the reaction proceeds to completion in ~5 min. The much faster reaction times may

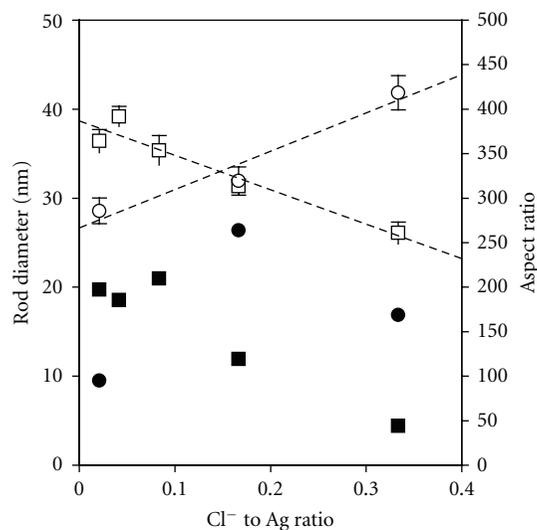


FIGURE 3: Change in Ag rod diameter and rod aspect ratio as a function of MnCl_2 and MgCl_2 concentration. Data for MgCl_2 is shown in the open and closed squares while that for MnCl_2 is shown in the open and closed circles. Open symbols are the mean rod diameters (error bars are the standard error of the mean), plotted on the left axis, and closed symbols are the aspect ratios, plotted on the right axis.

not allow for oxidative etching to occur at a rate comparable to growth. FeCl_3 resulted in relatively short rods with a diameter of 57 nm and an AR on the order of 9. CuCl_2 resulted in similarly short large-diameter rods with an AR on the order of 8 to 23 depending on concentration of the salt. In contrast, MnCl_2 resulted in relatively small-diameter rods with AR on the order of 160–260 and of the three transition metals evaluated seems the most suitable for the production of high aspect ratio Ag nanowires using the microwave-assisted process. The use of MnCl_2 results in rods with smaller diameters and longer lengths than NaCl at a similar yield.

To further explore the effect of changing the salt concentration on the rod diameter, several more concentrations of MgCl_2 and MnCl_2 were evaluated. Figure 3 shows the mean rod diameter obtained as a function of the Cl^- to AgNO_3 ratio for both MgCl_2 and MnCl_2 . In the case of MgCl_2 , increased concentration resulted in smaller-diameter rods. However, the rod length was significantly reduced as well resulting in a reduction in AR from 200 to ~ 50 over the concentration range evaluated. Different results were obtained when the concentration of MnCl_2 was changed. Smaller-diameter rods were obtained at the lowest salt concentration while the rod length and, thus, aspect ratio peaked at the intermediate concentration.

3.2. Effects of Changes in Procedure. Others have found that small changes to the reaction conditions can have significant impacts on the formation of nanoparticles [3, 5–8]. Here we report some effects of reaction conditions on the formation of silver nanorods and wires that we noted during the development of the procedure used to identify the effects of the cation used as reported above. During this development,

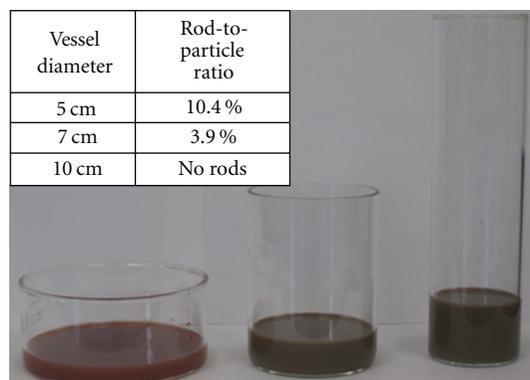


FIGURE 4: Comparison between three container shapes evaluated in initial work. The containers total volume was the same, 300 mL, in all cases.

we noticed two experimental parameters that seemed to have a significant effect on rod formation in the microwave-assisted synthesis process, pre-reaction conditions, and the shape of the reactor used.

Table 2 shows typical results for five different reaction conditions that were evaluated. The reaction conditions that were varied were the method of mixing the stock solutions together in the reaction vessel, how long this mixing occurred whether or not the solution was purged with N_2 to remove dissolved O_2 , and how soon the microwave heating was initiated after all mixing and purging were completed. Method I was the method ultimately selected to prepare the data shown previously on the influence of salt selection. Increasing the mixing time from a few quick swirls to 2 minutes seemed to increase the yield and the length of the rods formed. Sonication in contrast results in smaller-diameter shorter rods. Eliminating the N_2 purge had little effect. However, simply waiting for 10 minutes before starting the microwave had a significant effect on the reaction. No rods were formed; only spherical particles and aggregates were produced.

To the best of our knowledge, there has not been any previous work on the effect of shape of container on the synthesis and yield of the silver nanoparticles. Reaction vessel's size and shape are rarely specified in published methods: Gou et al. used a "flask" [25], Chen et al. used a "three necked flask" [26], and Korte et al. used a "glass vial" [23]. Since the reaction is very temperature sensitive and different container shapes provide a different surface-to-volume ratio and heating path, it was hypothesized that container shape may influence results. Three containers were evaluated. All containers had the same wall thickness and were blown from the same glass. All containers had the same 300 mL brim full volume and a cylindrical shape but different masses and diameters. The mass of the containers shown in Figure 4 were 146.9 g, 122.8 g, and 110.9 g for the 5 cm, 7 cm, and 10 cm, respectively. The number ratio of rods to spheres, as determined by optical microscopy, is provided in the inset table of Figure 4. When the vessel with the largest diameter, thus the largest surface area, was used, no rods were found. As the container diameter was decreased, the number of rods increased. To ensure that these results were not simply due to

TABLE 2: Rod diameter, length, and number of rods produced for several reaction conditions examined. All samples were prepared using KCl as the salt with a Cl:AgNO₃ ratio of 1:6.

Method	Mixing	Mixing time (min.)	N ₂ gas (min.)	Microwave start time (min.)	Diameter (nm)	Length (μm)	Nominal AR	No. of rods
I	Hand	0.25	1	0	35.5 (6.4)	9.3 (1.4)	261	50 (6)
II	Hand	2	1	0	33.7 (8.4)	10.1 (2)	300	64 (8)
III	Sonication	2	1	0	30.1 (5.8)	7.3 (1.6)	243	42 (3)
IV	Hand	0.25	0	0	34.3 (5.4)	8.9 (2.7)	259	47 (17)
V	Hand	0.25	1	10	42.3 (17.3)	NA	NA	None

the irradiation level not being consistent within the microwave cavity, the reaction was repeated multiple times using the large-diameter vessel at various locations within the cavity. Since rods were not produced at any location when the large-diameter vessel was used, uneven power distribution in the microwave chamber was not the cause of the lack of rod formation. To see if the differences were due to evaporation from the larger surface of the large-diameter containers, a watch glass was used to reduce evaporation from all containers. This did not affect the rate of rod formation. The nanorod-to-nanoparticle ratio for the smaller-diameter vessels remained similar, while the large-diameter vessel failed to produce rods.

Several factors likely contributed to the results. First, the surface-to-volume ratio of the reaction mass may affect both the heat distribution within the volume at short times (before convective heat transfer starts) and the cooling rate of the reaction mass after removal from the microwave. The change in total mass of glass is an increase of ~35% going from the highest-diameter to the lowest-diameter vessel used in this study. Tests in which reaction was stopped at defined times and the temperature of the dispersion measured with a fast response time thermocouple failed to show any differences in peak reaction temperature or heat up rates between the three reaction vessels evaluated. However, it was noted that the increased free surface area samples resulted in increased cooling rates after the reactor was removed from the microwave. Therefore, cooling rate and the progress of the reaction during cooling may be a factor. Second, vessel geometry affects how the initial reaction is distributed throughout the reactor volume. As the irradiation is attenuated by absorbance by the reactants, the power level drops from the outside of the reaction volume towards the centre of the reactor. This type of temperature differential would not have been detected in the measurements of overall heat up rate performed. One might expect that a more even heating would result in more rods as the processes is nucleated equally everywhere within the reaction volume. However, the container that should have the most even heating of the reactants and the large-diameter vessel resulted in the lowest production of rods. While the source of the effect of container geometry on nanorod yield is not clear, these results highlight that container shape, a parameter not typically included in published methods, can profoundly affect results. The importance of stating container shape in published methods should not be overlooked.

4. Conclusions

A range of salts were tested in the microwave-assisted polyol synthesis of silver nanorods. The results indicate that the behaviour of these salts in the microwave-assisted process is different than in reactions in which the heating is conducted over a longer time. Most notably iron and copper salts, which have been shown to dramatically improve the production yield and aspect ratio of silver nanorods produced in the traditional process, were shown to reduce the yield and length and significantly increase the diameter of the rods obtained in the microwave-assisted process. Changing the concentration of manganese chloride and magnesium chloride was a suitable route to control the aspect ratio and diameter of the produced nanorods. In particular manganese chloride can be used to produce rods with similar yields to sodium chloride but with smaller diameter and longer lengths, thus, AR ~30% higher. The effects of these two cations are directionally different on the rod diameter. These results suggest that, while the microwave-assisted synthesis process is a facile route to nanorods the roles of cations are fundamentally different than those in the traditional polyol process.

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