

## TITANIUM DIOXIDE NANOPARTICLES BY ADULTRATION EMULATION PROCESS

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### Abstract

A diverse range of consumer products contain silver nanoparticles. These products contain antibacterial/antifungal agents. Few examples of such products are air sanitizer, respirators, wet wipes, detergents, soaps, shampoos, toothpastes, air filters, coatings of refrigerators, vacuum cleaners, washing machines, food storage containers, cellular phones etc (Buzea 2007). The silver particles in nano-scale exhibit high-antibacterial activity and have no intolerable cytotoxic effects for human beings.

**Keywords:** Nanoparticles, Emulation.

### Introduction

Nanoscience is a scientific effort towards achieving complete control over of atoms, molecules and larger atomic structures including surfaces and bulk material. This control at the most basic level does not, however, come without difficulty, and at this point basic science is struggling to understand even the simplest building blocks and how they interact. Once this understanding is secured, nanotechnology will be apt to affect every aspect of human life, from the way we produce energy to the way we cure diseases. The basis of all life is molecular motion. As the great physicist Richard Feynman (Feynman, Leighton et al. 1995) said

*“If, in some cataclysm, all of scientific sentence passed on to the next generations of creatures, what statement would contain the most information in the fewest words? I believe it is the atomic hypothesis (or the atomic fact, whatever you wish to call it) that all things are made of atoms –little particles that move around in perpetual motion, attracting each other when they are a little distance apart, but repelling upon being squeezed into one another. In that one sentence, you will see, there is an enormous amount of information about the world, if just a little imagination and thinking are applied.”*

Controlling the physical and chemical properties of materials requires detailed knowledge about the behavior of the atoms and their interplay with other atoms in their surroundings. It also requires materials that will allow the manipulations to result in a broad range of properties. Metal oxides are proving to be a very interesting group of materials in this respect, because they cover the entire range of properties available; some are high superconducting while other are insulators, some are magnetic others not, and both their optical and mechanical properties vary a great deal.

### Elementary Features

#### Synthesis of Nano Materials

We can synthesize nanoparticles by two different methods: by downscaling i.e. by making things smaller, and by upscaling i.e. by constructing things from small building blocks. The first method-own” is, and called the second method “topis called “bottom-up” approach. The top-down approach follows the general trend of the microelectronic industry towards miniaturization of

integrated semi-conductor circuits. The lithographic techniques (top-down) offer the connection between structure and technical environment. Top-down approach involves typical solid-state processing of the materials. These methods are based on the reduction of bulk (micro) sized materials into the nano-scale. High energy ball milling or micro fluidizers are used to break down dispersed solids to 100 nm. Coarse-grained materials (metals, ceramic, and polymers) in the form of powders are crushed mechanically in ball milling by hard materials such as steel or tungsten carbide. This repeated deformation due to applied forces can cause large reduction in grain size since energy is being continuously pumped into crystalline structures to create lattice defects. However, this approach is not suitable for preparing uniformly shaped materials, and it is very difficult to realize very small particles even with high energy consumption.

### Different methods for synthesis of Silver and TiO<sub>2</sub> Nanoparticles

#### 1. Synthesis of silver Nanoparticles by different processes

With infinite applications in almost every field, nanotechnology is growing and becoming popular in academia and industry. Nanomaterials have attracted considerable interest due to their peculiar characteristics such as optical, mechanical, electronic and magnetic properties. The synthesis of noble metal nanoparticles has been a subject of numerous applications.

Over the last decades silver has been engineered into nanoparticles, structures from 1 to 100 nm in

size. Owing to their small size, the total surface area of the nanoparticles is maximized, leading to the highest value of the activity to weight ratio. The ancient Greek and Roman civilizations used silver vessels to keep water potable. Since the nineteenth century, silver based compounds have been used widely in bactericidal applications in healing of burns and also in wound therapy (H.Klasen 2000).

Furthermore, currently a diverse range of consumer products contain silver nanoparticles. These products contain antibacterial/antifungal agents. Few examples of such products are air sanitizer, respirators, wet wipes, detergents, soaps, shampoos, toothpastes, air filters, coatings of refrigerators, vacuum cleaners, washing machines, food storage containers, cellular phones etc (Buzea 2007). The silver particles in nano-scale exhibit high-antibacterial activity and have no intolerable cytotoxic effects for human beings. The antibacterial effect has been tested for yeast and *E. coli* by (Kim, Kuk et al. 2007). The experimental results showed that the growth inhibition effect of silver nanoparticles was in a concentration-dependent manner. They concluded that the silver nanoparticles were applicable to diverse medical devices and antimicrobial systems.

#### Low Angle Laser Light Scattering (LALLS)

Low angle of laser light scattering is also called as laser diffraction. It collects light scattered from particles in a collimated laser beam by an array of detectors in the focal plane of the collecting lens as in Figure. The angle varies from 14° for the early instruments up to 40° for the recent ones. The method is based on Fraunhofer diffraction theory as explained before.

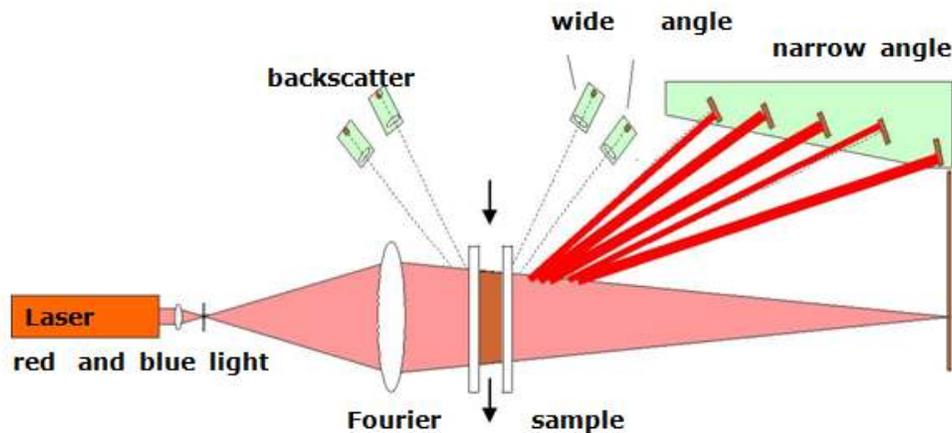


Figure : Schematic principle of Mastersizer 2000

Mastersizer 2000 is a commercial instrument from Malvern Company. It uses the technique of laser diffraction to accurately, quickly and reliably determine the size of particles from 0.02 to 2000 μm. The system can analyze emulsions,

suspensions, and dry powders in few seconds only without prior calibration. MS 2000 is a multifunction apparatus, since it can measure particle size, structure, and specific surface area simultaneously.

Two different models are used essentially in Mastersizer as a combination between Fraunhofer diffraction and Mie scattering theory. Fraunhofer approximation covers measurement for large particle while Mie theory predicts about all particles, small or large, transparent or opaque. Mie theory allows for primary scattering from the surface of the particle and also for the secondary scattering caused by light refraction within the particle (see Figure ). Mastersizer 2000 applies three kinds of detectors to collect the total scattering intensity as a function of angle. They are: (1) wide angle detectors, to grasp low scattering intensity from fine particles; (2) narrow angle detectors contained in focal plane optics to detect high scattering intensity from large particles; and (3) backscatter detectors. It also uses dual wavelength of light which are (He-Ne at  $\lambda=633$  nm) and blue light ( $\lambda=466$  nm) instead of only one wavelength to accommodate high resolution of measurement. The schematic principle of Mastersizer 2000 can be seen in Figure 3.10 while the commercial instrument is depicted in Figure 5



Figure: Mastersizer 2000

## Research Methodologies

This chapter elucidates the synthesis of silver nanoparticles by using capping agent along with reducing agent. The second part deals with synthesis and surface stabilization of sol-gel TiO<sub>2</sub> nanoparticles by means of sol-gel process with different surfactants.

This chapter contains experimental procedures, scientific data of the materials, capping agents, surfactants, and reducing agents. A list of chemicals and solvents used is also included in this chapter

## Experimental Set up

This section demonstrates and explains the experimental methodology and procedure for

Chemical name	IUPAC name	Formula	Source	Concentration %
Silver nitrate	Silver nitric acid	AgNO <sub>3</sub>	Alfa Aesar	99.9 %
Trisodium-citrate	Trisodium2-	C <sub>6</sub> H <sub>5</sub> Na <sub>3</sub> O <sub>7</sub> ·2H <sub>2</sub> O	Alfa	97.0 %

producing oxide and noble nanoparticles under the chemical reduction and sol-gel precipitation process.

## Types and Characteristics of Stirrer

The focus will now be on the apparatuses commonly used for producing and measuring particle sizes, structures, and shapes. Nanoparticles are usually generated inside stirred tank reactor or vessel cylindrical in form with a vertical axis. A standardized design of a vessel is similar to Figure, however detail design depends on the requirement of different situations. Impeller, heater or cooler in form of jacket or thermostat, thermometer device, baffles, speed control and drain valve are the accessories provided. A motor drives an impeller, which is mounted on an overhung shaft. The impeller causes the liquid to circulate through the vessel and eventually return to the impeller. While all of this happens, the homogeneous system in the vessel is maintained.

## Silver nanoparticles synthesis

Noble nanoparticles have been extensively investigated because of their unique electronic and optical properties that are different from bulk materials. In this section, synthesis of silver nanoparticles is done using bottom-up approach. In comparison with a top-down approach, bottom-up approach gives the advantage of producing stable silver nanoparticles, by the formation of defined crystalline nanoparticles structures. Normally a dilute solution of metal salt, surfactant and reducing reagent leads to the formation of clear golden-yellow colloidal solution by a bottom-up approach

## Experimental Method for Silver

The present work explores the formation of uniform silver particles through the reduction of silver nitrate with sodium formaldehyde sulfoxylate (SFS). Here sodium citrate was used as stabilizer as well as capping agent, and methanol was used to increase the dispersion of silver nanoparticles. All chemical substances used for this work are shown in Table.

Table List of chemicals used

dihydrate (Na-Citrate)	hydroxypropane1,2,3- tricarboxylate dihydrate		Aesar	
Sodium- formaldehyde sulphoxylate (SFS)	Sodium hydroxymethanesulfinate	CH <sub>3</sub> NaO <sub>3</sub> S	Alfa Aesar	97.0 % (dry wt.)
Methanol	Methanol	CH <sub>3</sub> OH	Merck	100%

*IUPAC: International Union of Pure and Applied Chemistry nomenclature*

## Results and Analysis

In this chapter, we discuss: first the synthesis of silver nanoparticles by double reduction method and second the agglomeration and disintegration process of titanium dioxide nanoparticles synthesized by sol-gel process.

The prime goal is the optimization of nanoparticles formation process in the liquid phase with different conditions. Silver and titanium dioxide nanoparticles are produced in the batch reactor. They are investigated both by experimentally as well as by simulations based on the population balance equations. The population balance models for agglomeration and disintegration leads to a system of integro-partial differential equations, which can be numerically solved by several numerical schemes. Here the cell average technique is used to solve PBEs and predict the particle size distributions and moments.

## Experimental results of silver nanoparticles

Synthesis of silver nanoparticles is done by double reduction method. In this process silver particles are capped with citrate ions and then it is reduced by sodium formaldehyde sulphoxylate. In general surface capped silver powder can be effectively converted to colloidal state via re-dispersion. Here, the agglomeration process is caused by rapid collision of the particles and their afterward bonding. Depending on their interactions, this collision results in the agglomeration or redispersion of particles. During the time of the process, after the drop wise addition of reducing agent the redispersion begins and then the particle size distribution develops rapidly. The size of the particles distribute into the wide and varied range. Particle size distribution and zeta potential were measured using Dynamic Light scattering method (DLS). Results obtained with all the experiments performed are summarized in tables and graphical representations in this section.

## Conclusion

Surface stabilized spherical titania particles have been synthesized in this study via the sol-gel process. Titanium tetra isopropoxide was used as a precursor. Three different surfactants were used for the synthesis of spherical titania particles of variable sizes. The particle size distributions were measured by the dynamic light scattering technique. The results from dynamic light scattering showed that the different stabilizers lead to entirely different particle size distributions. It has been shown that size and dispersity of colloidal particles can be controlled by appropriate choice of surfactants and polymers or salt that is added during the synthesis.

All experiments showed that after some time particle size distribution reaches a steady state. In the initial phase of experiments, large particles are observed, due to agglomeration process. Disintegration of agglomerates becomes more significant as the agglomerates become larger; it slows down the growth of agglomerates and creates smaller particles. Steady state condition is reached as the two opposing mechanisms balance each other. Application of various shear rates i.e., ( $\dot{\gamma} = 370, 623, 960, 1342 \text{ s}^{-1}$ ) to the reaction condition gives a tendency in which the higher the shear rates, the lower is the particle size distributions. Among the entire applied shear rates,  $\dot{\gamma} = 1342 \text{ s}^{-1}$  has been determined as the optimum shear rate for generating the smallest titanium dioxide nanoparticles.

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