

Controlling the nanoparticle size of silica in an acidic environment by using a strong magnetic field and a modified sol-gel techniques

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Abstract

In this study, we were able to create highly dispersed silica nanoparticles with diameters of less than one nm by changing the sol-gel technique. During the poly-condensation process, a strong magnetic field was applied to the silica sol to control particle size. The size of silica nanoparticles has a substantial impact on preparation elements such pH, magnetic field intensity, and exposure time. These parameters can be changed in a systematic manner to reduce or increase particle size. A dynamic light scattering test was also used to investigate the effect of a magnetic field on the particle size and dispersion of silica dust. Despite the fact that silica is naturally diamagnetic, the magnetic field has a considerable impact on their size growth. Magnetic fields altered the typical influence on silicon structure, resulting in crystal formation in the silicon sample under consideration. Many applications require small particle sizes and/or a narrow particle size dispersion. The building blocks of nanotechnology are usually made of low-dimension particles. The experts concluded that additional research into such strange phenomena will be required in the future.

Keywords: Acidic; dynamic light scattering; magnetic field; silica nanoparticle; sol-gel.

1. Introduction

The sol-gel procedure is one of the most well studied and standard methods of synthesizing silica nanoparticles to date. [Battisha, I. K *et. al.*, 2010, Sögaard *et. al.*, 2018]. Since Stöber 1968, [Stöber *et. al.*, 1968] there are plenty of particulate studies on the preparation of silica nanoparticles and nanoparticles size controlling. Significant consideration has been drawn to the effect of a magnetic field on the self-assembly behavior of molecules [Quickel *et. al.*, 2010 – Hirota *et. al.*, 2008], The nucleation and growth process of magnetic materials as well as of magnetic materials [Beecher *et. al.*, 2005, Yildiz *et. al.*, 2006]. However, there seems to be a lack of research in the literature covering a relationship during the sol-gel phase between the applied magnetic field and the resulting scale of diamagnetic nanoparticles.

Predominately, the critical parameter of colloidal systems is their particle size. The information about the size and shape of particulates is critical in predicting the initiate and growth of particulates. The geometry of the nanoparticle has a major effect on changes in properties, as it can influence both the surface-to-volume ratio and the surface energy ratio [Alattar, 2021; Odegard *et. al.*, 2005]. In order to impact high-technology applications, involving particular properties, imposed processing techniques enabling the regulation of the distribution of nanoparticles. The primary importance of effective control of morphology is that it provides open avenues for the prior prediction/optimization of material structures and properties [Zeng *et. al.*, 2008].

1.1 Particle growth mechanism

Based on the sol-gel process [Brinker *et. al.*, 1990], and throughout the sol stage linear structures are shaped, which then converted into nanoglobules, the consistency of which is pH dependent. Its nanoglobules form evenly throughout the sol and then rise in size during the last stages of development, as required by diffusion-limited aggregation [Julien 1989]. The aggregation started via the addition of an accelerator, such as catalyst, to the silica sol and the mechanism of aggregate and destabilization configuration somewhat demonstrated by the Derjaguin–Landau–Verwey–Overbeek (DLVO) theory. Furthermore, the sol stability will be discussed via the slow versus fast aggregation process. [Trompette *et. al.*, 2013– Sandkühler *et. al.*, 2003]. DLCA stands for diffusion-limited cluster aggregation, while RCA stands for reaction-limited cluster aggregation (RLCA).

1.1.1 Magnetic field influence

Strong magnetic fields, on the other hand, are frequently employed for comprehensive material characterization in a broad sense in proportion to the energy density of the applied magnetic field. The energy of interaction (Zeeman energy) between the material and the magnetic field should be small enough [Motokawa 1995]. When a high magnetic field is applied to a crystal during its solidification process, the orientation effect has a significant impact on the crystal's shape. A magnetic field induces a magnetic moment in a diamagnetic substance that is in opposition to the magnetic field that activates it. The classical explanation of diamagnetism regarding Lenz's law is not entirely accurate, where it is a quantum mechanical phenomenon [Stephen 2001]. More novel nanoparticles, however, have recently been studied, with major promises for contemporary physics and instrument applications [Tyagi *et. al.*, 2017].

Up to now, best of knowledge, this study is the first specific study reports the effect of applying a magnetic field on controlling the size of primary silica nanoparticles suspended in the sol phase.

2. Experimental.

2.1 Materials.

Tetraethylorthosilicate (TEOS > 99.0 % purity), spectroscopic grade and ethyl alcohol (200 proof, > 99.5 % purity), were obtained from Sigma Aldrich. Ammonium hydroxide (28-30 % concentration) supplied by BDH, and Ameresco supplied hydrochloric acid (0.15 M, > 99.0 % purity).

2.2 Procedure.

In silica sol preparation, condense silica (CS), TEOS and EtOH used as precursor and solvent, respectively. Hydrochloric acid was added to induce the hydrolysis of the precursor at a molar ratio of TEOS: EtOH: HCl (0.01 M) of 1:6.6:0.18, respectively. The pH of the final solution was 2.5, where this recipe denoted as the recipe (I). The irreversible poly-condensation reaction took place, under the presence of a magnetic field, via drop-wise adding of diluted NH_4OH (1.25 mol l^{-1}), as a base catalyst. Firstly, the pH of CS was converted from 2. to 3, with vigorous mixing using a glass rod. The samples kept under magnetic field for 5, 15, 30, 60 and 90 min. Secondly, using two magnetic field sources; 0.5T and 3T, at a fixed exposure time of two minutes, three samples of recipe (I) were studied by DLS. The pH value of these samples varied from 2 to 3, 4, and 5. Another CS recipe was prepared using different TEOS: EtOH: HCl (0.03 M) molar ratio of 1: 3: 1.2, respectively. This recipe denoted as (II) with pH of 2.5. The same poly-condensation procedure, of different pH, was repeated for this recipe as well.

2.3 Characterization

All samples filtered with 0.22 Super-pure syringe filters, otherwise will be mentioned. Then DLS experiments were done by using Zetasizer Nano-S instrument from Malvern using 632.8 nm laser and measured at the scattering angle of 173 degrees. Ethanol adopted in the DLS test as a dispersion solution.

XRD was performed using a Panalytical X' Pert PRO Alpha-1 XRD via $\text{Cu K}\alpha$ radiation. Due to the liquid nature of the sample, a few drops of each sol were poured on glass substrate then left to dry at room temperature. A light portion is crushing from coated silica as a powder, then used for XRD test.

3. Result

Table 1 shows the results of magnetic field exposure time effect on the silica particle size at pH= 3. The magnetic field showed fast influence on the silica particle size suspended in ethanol. Where, under the existence of a strong magnetic field of 3T, applied for two minutes, the poly-condensation process yielding a reduction in the particle size from 3.36 nm to 1.13 nm according to DLS result, based on particles number method. On the other hand, when the magnetic field exposure time increases for further periods, the silica particle size increased to their initial size range before introducing to the magnetic fields.

Table 1. Influence of magnetic field exposure time on the silica particle size at pH= 3.

Magnetic field (T)	0		3			
Time (min)	0	5	15	30	60	90
Particle size of recipe I (nm)	3.36	1.13	4.28	3.23	3.08	4.00

Under the presence of a high magnetic field, the sol pH value variations showed significant influence on the silica particles size. A dramatic divergence in the particle size was noticed through a slight change in the pH of the silica solution as given in table 2.

Table 2. Influence of magnetic field and pH values on silica particle size at an exposure time of 2 min. The gel time varied from two days to several days, and the sols still stabled for more than one year.

	Magnetic field status					Recipe No.	Magnetic field strength (T)	Dispersant media
	Non		Applied					
	pH 2.	pH 2.5	pH 3	pH 4	pH 5			
particle size (nm)	3.22	3.21	1.16	588*	261*	I	3	Ethanol
Final state	Sol	Sol	Sol	Gel	Gel			
particle size (nm)	3.22	3.21	622*	2.27	0.51	I	0.5	Ethanol
Final state	Sol	Sol	Gel	Gel	Sol			
particle size (nm)	30.1	30.3	5.68	28.1	67.5	II	3	water
Final state	Sol	Sol	Sol	Gel	Gel			

* Samples test without filtering

Figure 1-a and figure 2-b demonstrate the effect of pH value changing on the particle size of recipe (I) silica colloid, which was poly-condensed under two different magnetic field of 0.5 T and 3 T, respectively. The high matching between the three testing methods may confirm the particle size systematic dependence in the polycondensation process. Figure 1-c illustrates the pH value effect on the particle size of recipe II silica colloid. This recipe was poly-condensed under a magnetic field of 3 T.

The idea indicates a rise in suspension stability with increased surface load and decreasing salt levels trends in most loaded particle suspensions. The aggregation rate constants, often within a 2 factor and better, are predicted quite well in the quick regime [Bergström1997, Kobayashi *et. al.*, 2005].

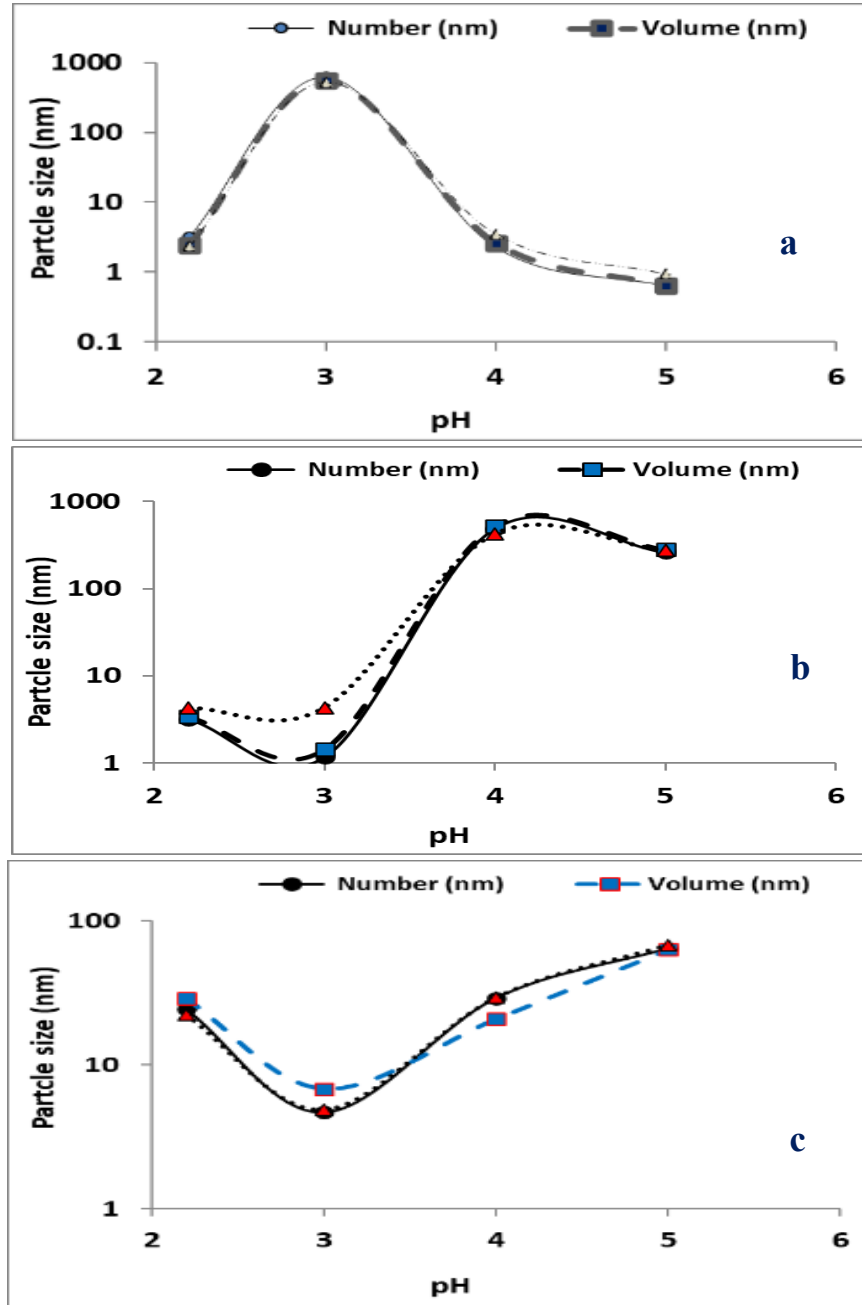


Fig. 1. Particle size results following three testing methods; number, volume, and intensity (a) recipe (I) at 0.5 T (b) recipe (I) at 3 T, (c) recipe (II) at 3 T.

Figure 2 illustrates the distribution of particle size by the number of recipes from the DLS graphical results and show (I) prepared under 3T (a) pH = 2 (b) pH = 3, (c) pH = 5, and under 0.5 T (d) pH = 2.5 (e) pH = 3 (f) pH = 5. Showing different distribution of particle size with different magnetic field the size of the silica particles is smaller than the domain size range (DFMN).

When the temperature rises over the so-called blocking temperature, each particle behaves like a gigantic paramagnetic atom and displays superparamagnetic conduct.

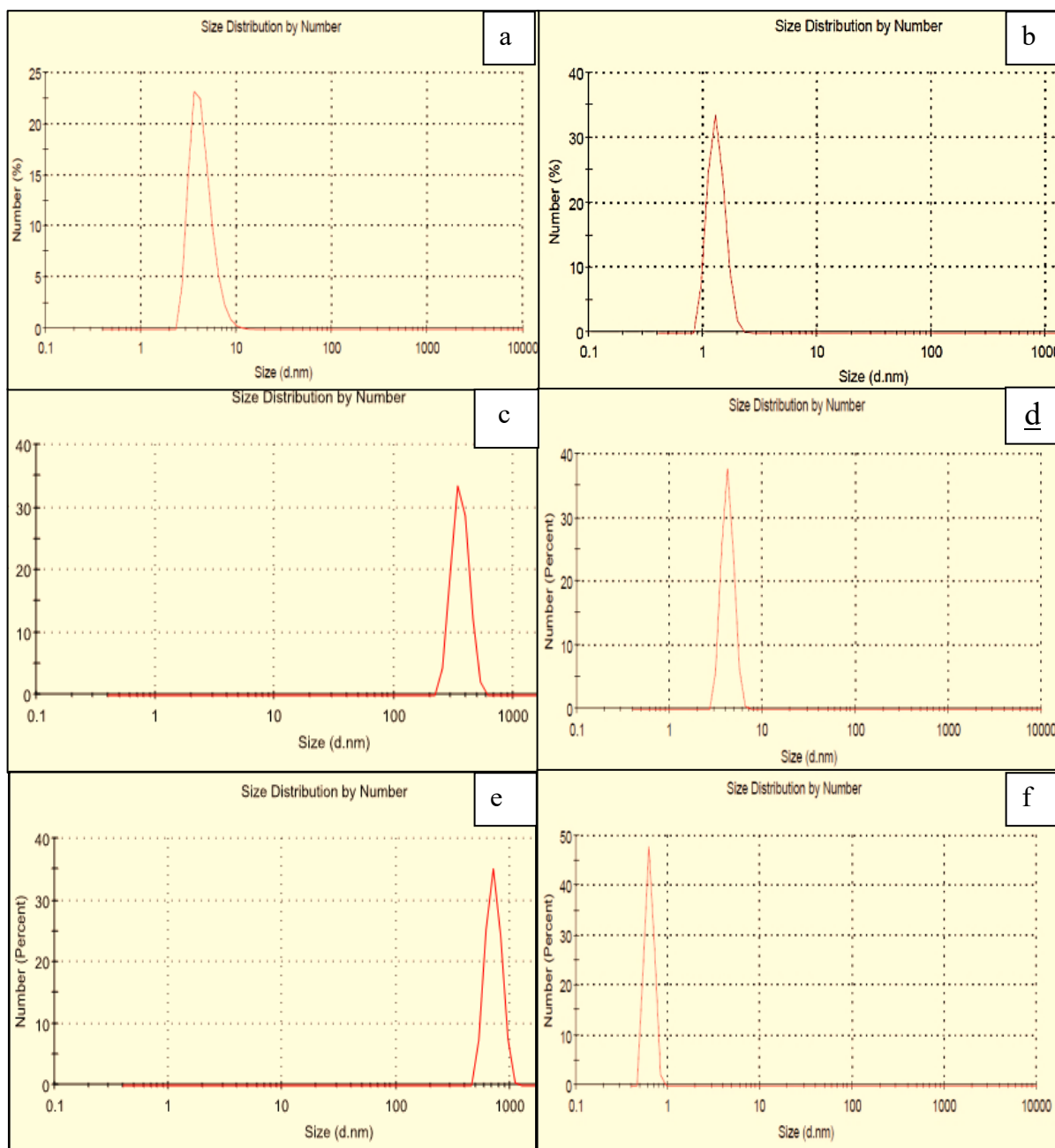


Fig. 2. DLS graphical results show the particles size distrebuion by number for recipe (I) prepared under 3T (a) pH = 2.2 (b) pH = 3,(c) pH = 5, and under 0.5 T (d) pH = 2.2 (e) pH = 3 (f) pH = 5.

4. Discussion

According to the availability of many acidic silica sols for the influence of a magnetic field, it was systematically worked out the route through the odd circumstances that may affect the silica sol. Theoretically, the strong magnetic field drifted the orientation of magnetic dipoles and or raises an energy splitting between the spin states of electrons and protons. As magnetic fields affect charged

particles, therefore, it is possible to force H^+ from one part of the solution to another, causing the local concentration of H^+ to increase instantly. Fundamentally, the pH measured as $-\log [H^+]$, therefore, by changing the concentration of H^+ , the pH will change. However, this effect will be incredibly small, particularly in non-ferric liquids, and thus hardly noticeable. Furthermore, we can, with full conviction, exclude this scenario, where the sol was continuously mixing during the magnetic field exposure. As a result, like those drifts do not affect varying the acidity of the sol.

Commonly, in DLS test, particle size distributions are calculated regarding particles volume, particles number or scattering intensity. The three methods were adopted; however, they show the same behavior, with a minor variation in the particle size values. Because the DLS technique generates distributions with intrinsic peak growth, there will be a continuous inaccuracy in the depiction of the intensity distribution. The volume and number distributions produced from these power distributions are best employed for statistical applications or for estimating relative proportions [Alattar 2021].

Figure 4 illustrates the magnetic field exposure time effect on the size of silica nanoparticles. Apparently, at the first two minutes of the exposure time, the essential reducing in the particles size has occurred, then the particles size increases. The long exposure time of a strong magnetic field may enhance the locally induce dipole moment in these nanoparticles. Subsequently these polarized nanoparticles were electrically aligned resulting in relatively long (few nanometers) agglomerated chains. These chains may be recorded as bigger size particles in the DLS test.

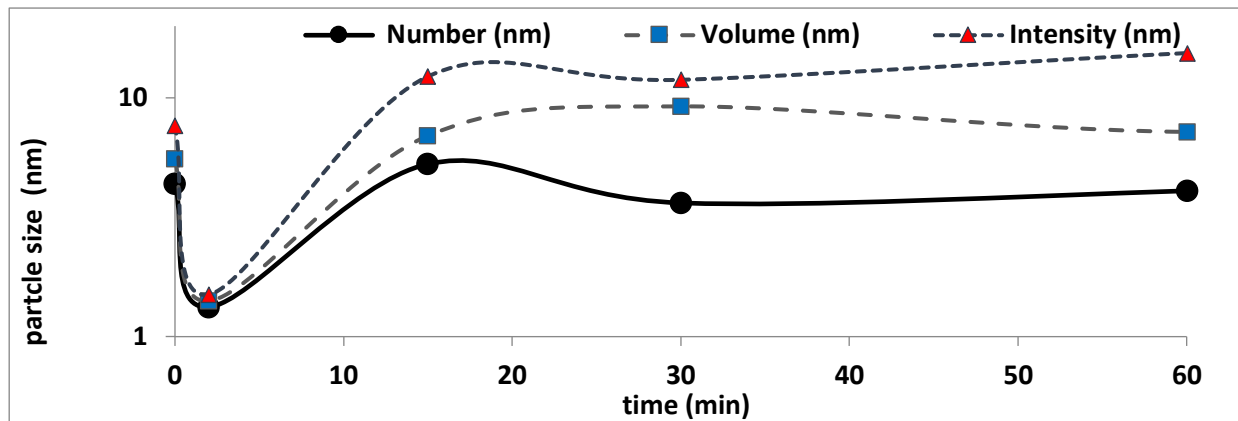


Fig. 4. Particle size results following three testing methods; number, volume, and intensity at different exposing time and pH = 3.

The applied magnetic field found to be of no noticeable effect on the particle size of the sample of $pH = 2.5$ (table 2), which cannot be classified based on the DLVO theory. How to understand the no response of the suspending silica nanoparticles to the high magnetic field. At the moment of zero charge for PZC, it would be logical to presume that the silica surface is unloaded. The behavior of these uncharged particles would also not be affected by the magnetic field. In this context, DLVO's theory defines particle suspension equilibrium by integrating electrostatic and van der Waals forces.

Van der Waals forces are weak outside of silica spheres [Bergström 1997]. This idea has received a lot of attention due to its failure to explain particle activity at lower pH values. Because there is no electrostatic repulsion, the silica particles remain stable at pH levels about 2. (the PZC). As a result of this contradiction, novel non-DLVO interactions, such as those that think the particle's water structure and the so-called "layer of gel," which is thought to exist at low pH, can interact, have been proposed. The stability of smaller nanoparticles at low pH levels may be explained by extra repulsive forces not described in the DLVO theory. Because they are mechanical barriers to particle aggregation and are only a few nanometers from the silica base, poly (silica acid) gel coatings are thought to be the source of these additional forces [Kobayashi *et. al.*, 2005, Johnson *et. al.*, 2008]. Because of its increased surface area, this gel coating has a similar effect on smaller nanoparticles as it does on larger nanoparticles. Because poly (silica acid) deteriorates at higher pH levels, the gel layer is only present at lower pH levels. These findings suggest that the DLVO theory needs to be complemented by studies that have knowledge of particle behavior. In the case of recipe (I), under the presence of high magnetic field 3T. The particles size decreased notably, from 3.22 nm to 1.16 nm when the sol pH value lightly increases from pH = 2.5 to pH = 3 respectively. This decreasing confirms that there is an influence of magnetic field on the particles size growth. Further increasing in sol pH value to pH = 4 will significantly increase the particles size from 1.16 nm to 688 nm, then at pH = 5 the particle size was being redacted to 261 nm.

Under a relatively low magnetic field, 0.5 T, the particles size increased dramatically, from 3.22 nm to 622 nm, when the sol pH value lightly increases from 2.5 to 3 respectively. Further increasing in sol pH value to pH = 4 will dramatically decrease the particles size from 622 nm to 3.27 nm, then at pH value of pH = 5, to 0.51 nm.

To the best of our knowledge, there is no theoretical model cover these odd behaviors. It may be that, if the intensity of the applied magnetic field is strong enough (for example 3T), it may snatch the attached water molecules from the silica surface. Whereas the silicone oxide-derived sol-gel networks produce mainly linear or arbitrarily branched polymers under acid-catalyzed conditions [Moon *et. al.*, 2017]. The strong magnetic field may attack then broke the weak points inside the polymerlike nanoparticles itself as well. At pH = 3, this attacking then the broking process will lead subsequently to tiny silica nanoparticles (from 3.22 nm to 1.16 nm). Taken into consideration the almost uncharged silica nanoparticles under pH = 3 environment. However, further investigations should be carried out so that to confirm this postulation.

At higher pH values, pH = 4 and pH = 5, the surface of the silica particles will be more charged. However, it can be concluded that soliton-type excitation occurs on the silica chain, for example, as polymers are used, and this excitation plays a role as an electrical conductor. Ring currents on loops produce magnetic moments perpendicular to loops in silica networks. The loops tend to align, thus, perpendicular to the magnetic field. Now, this may mean that the excitement is not limited to this type; there may be excitement around magnetic moments. In such case, parallel to the magnetic field, the silica chains are aligned. The crystallites were then oriented with their crystallographic axes in the direction of the magnetic field applied during the preparation of the light. The magnetic field applied during the preparation of the gel is thus supposed to make the structure of the silica particles anisotropic. Taking into account that there is an easy direction of

magnetization in silica particles, therefore, the reduction in magneto-crystalline anisotropy energy may drive the reduction of the silica particle size in the sol under application of a strong magnetic field. Furthermore, the applied magnetic field may build up a barrier around the silica particles preventing the grain growth process. Nano-magnetism, however, typically recognizes so-called single-domain particles; for single-domain size, the typical values range from 5 to 125 nm. All of these particles are domain-free magnetic nanoparticles because a single particle with a size equal to the minimal domain size isn't divided into domains. This means that particles are smaller than the range of domain sizes (DFMN). Each particle functions like a gigantic paramagnetic atom and manifested super-paramagnetic conductivity during the moment when the temperature exceeds the so-called blocking temperature. On the other hand, in case of the presence of relatively low magnetic field, 0.5 T, there was a reverse scenario. At a little shifting from ZPC, pH = 3, the presence of such magnetic field snatches the attached to the molecules of water from the silica surface. This snatching might cause the hydration layer to breakdown, allowing aggregation to occur, leading to a 622nm wide agglomerated particle. We should take into account that the silicon dioxide is diamagnetic material; basically, these materials have a little response for such relatively low magnetic fields. While at higher pH values, pH = 4 and pH = 5, the surface of the agglomerated silica particles will be charged. This sizable charged particles then polarized owing to the applied magnetic field, inducing inner electrical dipole moment. Therefore, the combination of the electric dipole moment and electrically charged sizable particles, as repulsive forces, maybe excesses the agglomeration adhesion force.

So this perturbation may detonate this sizable particle to tiny nanoparticles, 0.6 nm in size. The monotonically decreasing in the silica particle sizes according to the increases of pH value, Table 2, may confirm the influence of the detonating power suggested above. Take into account monotonically increasing pH values reveals an increase in surface charges then dipole moment resulting in high detonating power.

5. Conclusion

Applied of magnetic field throughout the preparation of the gel is thus supposed to make the structure of the silica particles anisotropic. Taking into account that there is an easy direction of magnetization in silica particles, therefore, the reduction in magneto-crystalline anisotropy energy may drive the reduction of the silica particle size in the sol under application of a strong magnetic field. Furthermore, the applied magnetic field may build up a barrier around the silica particles preventing the grain

growth process. Nano-magnetism, however, typically recognizes so-called single-domain particles; for single-domain size, the typical values range from 10 to 140 nm

However, the accurate monitoring of the scale of nanoparticles using this method has proven to be a challenging task and further investigations should be carried out in order to improve all experimental parameters.

Due to the large number of searches for influences that are important in controlling the size of nanoparticles, where many studies have used light such as laser and others, or heat, pressure, or electricity, but the magnetic field is also important and this is proven through this study, this study is modern in its kind and application.

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