Characterization and Transformation of Silicon Dioxide Nanoparticles in Aqueous Suspensions: Influence of pH

T. Y. Yee and M. Omar Fatehah*

School of Civil Engineering, Universiti Sains Malaysia, 14300 Nibong Tebal, Malaysia

P A P E R I N F O

A B S T R A C T

Silicon dioxide (SiO_2) in nanoscale had been detected as waste product in river water for the past two decades and it is recently proven to have adverse effects toward human and animal health, the ecosystem and water treatment system. The removal of SiO_2 nanoparticles (NPs) from water still remains a challenge due to its small size and unknown interactions within the water body. In this study, dynamic light scattering (DLS) technique was applied to characterize SiO_2 in terms of surface charge and particle size as a function of pH within the range of 2 to 11 to analyze the aggregation behavior and significance of the intermolacular interactions in deionized (DI) water and tap water. DLS analysis identified both pH values of the point of zero charge (pH_{PZC}) of SiO_2 NPs in DI water and tap water at pH 3.2 and pH 2.8 respectively. The initial pH was discovered at 7.1 in tap water with a mean particle size of 346 nm and an average surface charge value of -27 mV compared to initial pH of DI water which was 5.4 with mean particle size of 295 nm and an average surface charge value of -33 mV. It was found that both in DI water and tap water, SiO_2 NPs aggregated and increased in particle size but reduced in surface charge when pH slowly decreased towards their respective pH_{PZC} from the initial pH by adding 0.25M of hydrochloric acid. The mean particle size at pH_{PZC} in DI water is measured at 1750 nm larger compared to the mean particle size in tap water indicating that the presence of other ions in tap water suppressed the aggregation process. In conclusion, results suggests that pH does influence the surface charge of SiO_2 NPs and affect the stability behavior and its interaction processes in aqueous suspensions.

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INTRODUCTION

For the last two decades, nanotechnology industries has increased in production of nanomaterials, subsequently leading to an inevitably release of by-products made of these engineered nanoparticles (ENPs) into the environment. The unintentionally discharge of ENPs as waste into rivers and sea water where some of them already ending up in our drinking waters at detectable concentration levels [4, 13]. These nanoparticles in industrial effluents are able to enter and build up in the human body through skin contact, inhalation of gas released from water, direct ingestion of water polluted with nanoparticles, or indirect exposure from eating vegetables and aquatic life such as fish, shrimps and octopus as food [4].

The investigation of hazards towards humans and animals caused by the risks of nanoparticles is still ongoing and remains a challenge even though there are plentiful applications to be used [16]. However, there are properties of nanoparticles that might give negative effects to human body and animals. The very large surface area of these nanoparticles can generate harmful oxyradicals with other chemical substances; that can cause cytotoxicity. In addition, the nanoparticles have the ability to penetrate the body and cells thus provide potential ways for the delivery of nanoparticle-associated toxic pollutants to places where they would not go under usual condition [13]. As a result, nanoparticles can act as a vector for concentrated hazardous compounds [12].

In this study, the main focus of the nanoparticles to be investigated is silicon dioxide (SiO_2) in water. The main reason SiO_2 nanoparticles (NPs) is chosen because of the very large quantities of it is produced every year (10^2-10^3 ton/year) [11] compared to other categories and types of nanoparticles such as pure metallic nanoparticles, metallic oxides or oxy-hydroxides and carbonaceous nanoparticles. Although most of the contribution of SiO_2 NPs come from the discharged of semiconductor industries, SiO_2 NPs will be a major issue to environmental pollution and might give

* Corresponding author: Fatehah Mohd Omar
E-mail: cefatehah@usm.my

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negative effects to our health because it have been widely used in our daily lives as well since the dawn of nanotechnology as nanomaterials in food products, paints, cosmetics, and biomedical applications such as drug delivery and multimodal imaging. Although there are still lack of toxicological data about the danger possessed by SiO_2 NPs in food and drinks, there are more and more researches shown that it might have the toxicity. For example, it showed the potential adverse impacts on human gut in one of the research [21]. Currently, the only regulation related to safety of using silica in food is only established by U.S. Food and Drug Administration (FDA) under 21 CFR, which the silica content should not be more than 2 % of food weight.

Recently, SiO_2 NPs in water not only found out to be dangerous to human health, but also had brought negative effects towards ecology system, animal health as well as water treatment process. It was discovered that SiO_2 NPs affects the health and behavior of Zebrafish Embryos and Larvae [7] and causes renal and kidney disease to buffalo [18]. Because of their small size, SiO_2 NPs surpasses conventional water treatment processes thus make them hard to be removed from water. This is also due to nanoparticles have specific properties because of their surface area over volume ratio make them mix with other substances in water. It clogs water pipes [2] and cause low separation efficiency [12]. It is also one of the most common foulant for membrane fouling [22].

Therefore, the development of specific treatments (e.g. coagulation, flotation or filtration) and suitable additives for water consisting nanoparticles is necessary. In 2007, the treatment of CMP effluents contain silica nanoparticles by chemical coagulation with polyaluminium chloride (PAC) has been studied [3] but it causes a bulky sludge. In 2012, investigation of the coagulation and flocculation of nanosilica by some organic polymers had been carried out but the efficiency does not meet expected results [1]. In the same year as 2012, $AlCl_3$ was used as cationic surfactant but a lot of coagulants required to remove silica from water [11]. In 2013, aggregation by cationic surfactant cetyltrimethylammonium bromide (CTAB) had been investigated and appeared to have a better removal but nano-size smaller than 30 nm had found out to be difficult to remove [12]. Meanwhile, other researches had been done to remove SiO_2 from water such as coagulation process, reverse osmosis process, micro-buble flotation technology, electrocoagulation technology, suppression technique with chemical scale inhibitor and ion exchange technology but all the results remain to be less efficiency or uneconomically [24]. In summary, due to the separation of SiO_2 NPs from water is difficult and have major adverse impacts to environment and health, it is important to study the fundamental of nanoparticles life cycle in water before

efficiently removing them from the water. However, the fate of nanoparticles in actual aquatic environmental system is complex and hard to predict because of many factors. According to Zhang et al. [23], the transformation of ENPs in water is determined by three main root causes: (1) aqueous solubility; (2) possible chemical reaction of the nanoparticles with the aquatic system; and (3) their interaction with biological system in the environment. The three factors establish that the significant of the environment conditions ENPs had to react such as pH, ionic composition, ionic strength, salinity, natural organic matter content, liquid motion and other properties that may alter the characteristics of ENPs. However, the types and behaviour of ENPs are important to change the fate of ENPs as well [14]. Nanoparticle behaviour is based on its unique particle properties such as its dimensions, structure, chemical composition and zeta potential as well as state of particle whether it is free or matrix incorporated while discharge into the aquatic system [14]. After knowing the properties and behaviour of ENPs in different environmental conditions, the interactions of particles with itself or aggregate with environmental surface will be discovered and only then their fate in the environment can be anticipated [15]. In this study, solution pH had been selected among all those factors as the manipulated variable in order to identify the basic characteristic of SiO_2 NPs in deionized (DI) water and tap water by measuring the surface charge and particle size distribution.

MATERIAL AND METHODS

Sample Preparation
Silicon dioxide was purchased from R&M Chemicals, United Kingdom in the form of nanopowder with 99.0% purity and below 1000 nm diameter size. SiO_2 nanoparticle powder was added to a background solution (deionized water at >18.2 MΩ, Purelab Option-Q, ELGA) for DI water sample and (Tap water collected at Civil Engineering School of Universiti Sains Malaysia) for tap water sample to make a 100 mg/L solution for each experiment. Ultrasonication was then done using an ultrasonicator (Elmasonic E Ultrasonic Bath E30H) for 30 min to break up the dry NP aggregates in the solution. Analytical grade NaCl, NaOH and HCl were purchased from Sigma-Aldrich. The nanoparticle suspensions were titrated with diluted HCl and NaOH solutions and the pH was monitored using a pH meter (BP3001, Trans Instruments). An aliquot of the sample was taken out at specific pH values and the surface charge and particle size were determined with a ZetaSizer Nano ZS (Malvern) at 25 °C. Freshly made suspensions were prepared before each experimental run. Experiments were done in

pentaplicates and the mean surface charge and particle size were determined.

Surface Charge and Particle Size measurements
Surface charge of SiO_2 NPs in DI water and tap water was determined according to the Smoluchowski equation while the nanoparticle size was measured using the dynamic light scattering (DLS) method with the Zetasizer Nano ZS (Malvern). While measuring, the particle suspension had been passed through by a 633-nm He–Ne laser beam and the scattered light is detected and collected by a photo-detector at a fixed scattering angle of 173°. The correlator then collect the light signal and carry out a light scattering intensity autocorrelation function. The diffusion coefficients of the particles that undergo Brownian motion are defined by the fluctuation of the autocorrelation function over decay time. The particle size of SiO_2 is determined from the diffusion coefficients in terms of the Stoke–Einstein equation. The range of particles size that can be measured is limited in the range from 2 nm to 6 μm. Nanoparticle suspensions were injected into polystyrene vials using a syringe for DLS measurements. All vials were used only once and were rinsed with DI water before being filled with suspensions. Measurements were carried out for 5 min at room temperature (25 °C) to obtain the mean particle size.

RESULTS AND DISCUSSION

Characterization of SiO2 nanoparticle in DI water
In order to have a better understanding of the pH effect on SiO_2 surface charge and aggregation, a SiO_2 suspension (100 mg/L) was titrated with acid (hydrochloric acid) and base (sodium hydroxide) within the pH range of pH 2.5 to pH 11 in DI water (Figure 1a). When silica chemically reacted with water below pH 9, small amounts of silica dissolve and enter the aqueous phase and formed SiO_2 with a partial of surface consists of $Si(OH)_4$ which is silanol groups (Si-OH) as shown in equation below [19]:

$(SiO_2)_n + 2H_2O = (SiO_2)_{n-1} + Si(OH)_4$ (1)
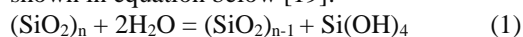
When the pH is higher than 9, it will start to form $(HO)_3SiO^-$ and $(HO)_2SiO_2^{2-}$ [9] as shown in equation below:

$Si(OH)_4 = (HO)_3SiO^- + H^+$ (2)

$Si(OH)_4 = (HO)_2SiO_2^{2-} + 2H^+$ (3)
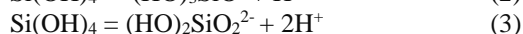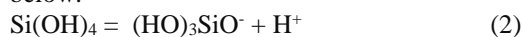
The initial pH of the SiO_2 suspension was determined at 5.4 with a surface charge between -31 mv and -35 mv and a mean particle size of 296 nm. When the pH was decreased, the surface charge was found to decrease rapidly from 5.4 to 4, then slowly to the point of zero charge (pH_{PZC}) which was measured at pH 3.2. This is important as other study have reported that pH 3 is close

to the point of zero charge for SiO_2 NPs [14]. As the pH was continue decreased from pH_{PZC} towards 2.5, the surface charge remain slightly positive in charge (< +5mv). Meanwhile, by increasing the pH above pH 5.4 and towards pH 11, the surface charge was affected and decreased slowly between -31 mv and -35 mv towards -59 mv and -61 mv.

The particle size of SiO_2 aggregates is plotted as a function of pH in Figure 1b. At an initial pH of 5.4, the mean particle size of SiO_2 measures at 296 nm. When the pH was decreased, the particle size was found to increase rapidly from 5.4 to 3.2, with the highest particle size around 5000 nm. It is proven that aggregation occurred during pH 5.4 to pH 3.2. As the pH was decreased from 3.2 to 2.5, the particle size decreases rapidly to 1000 nm. When the pH was increased from 5.4 to 11, the particles size remains below 500 nm. SiO_2 was in dissolution region when pH was higher was supported by the results obtained.

a)

b)
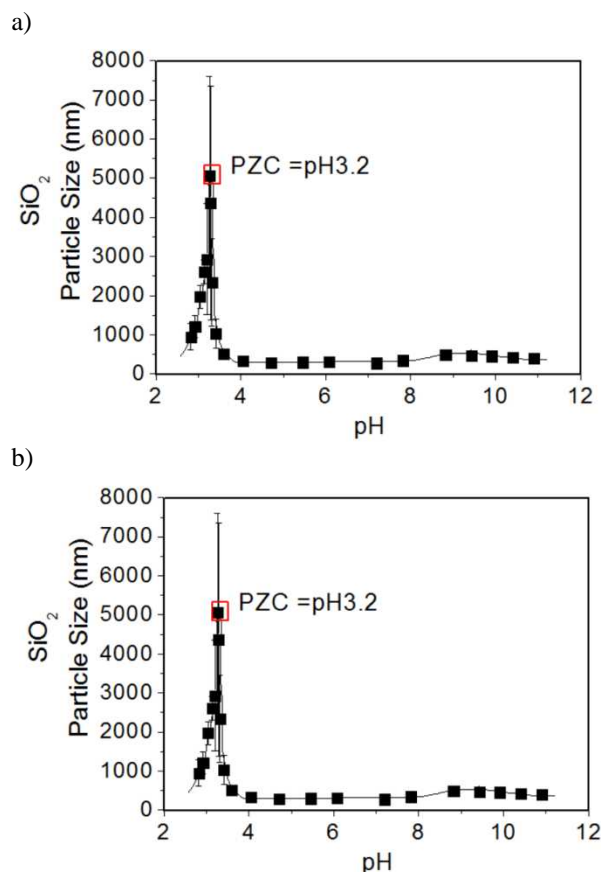
Figure 1(a) Surface charge of SiO_2 in DI water as a function of pH at a range of pH 2.5 to 11. The initial pH was found at 5.4 with a surface charge of -33 ±2 mv. The pH of point of zero charge (PZC) was identified at 3.2. **(b)** Particle size measurement of SiO_2 in DI water as a function of pH at a range of pH 2.5 to 11. The initial pH was found at 5.4 with a particle size of 296 ±19 nm. The pH of point of zero charge (PZC) was identified at 3.2.

The particle size measurements in Figure 1b are concurrent with the surface charge values presented in Figure 1a. The mean particle size was shown to be the highest when in the range of pH_{PZC}. Furthermore, the particle sizes were also found out to be smaller when the surface charge was further from the point of zero charge. At PZC, the silanol groups (Si-OH) were stabilize due to the same amount of negative and positive charges of silanol groups created on the surface of the silica particles. The "hairy layer" in Figure 2 which consists of polysilicilic acid on the silica particle surface was created at low pH with high ionic strength [11]. The "hairy layer" might be caused by water molecules diffusing into and becoming larger with the silica.
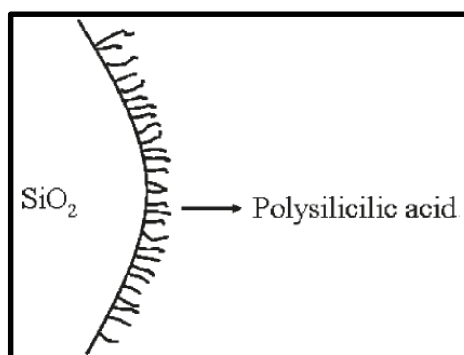
Figure 2 The schematic of the "hairy layer" on the silica particle surface [11].

Characterization of SiO₂ nanoparticle in tap water

In order to have a better understanding of the pH effect on SiO₂ surface charge and aggregation, a SiO₂ suspension (100 mg/L) was titrated with acid (hydrochloric acid) and base (sodium hydroxide) within the pH range of pH 2.5 to pH 11 in tap water (Figure 3a). The initial pH of the SiO₂ suspension was determined at 7.1 with a surface charge between -25 mv and -29 mv and a mean particle size of 346 nm. When the pH was decreased, the surface charge was found to decrease gradually from 7.1 to 4, then rapidly to the point of zero charge (pH_{PZC}) which was measured at pH 2.8. This is important as other study have reported that pH 3 is close to the point of zero charge for SiO2 NPs [14]. As the pH was continue decreased from pH_{PZC} towards 2.5, the surface charge remain slightly positive in charge (< +5mv). Meanwhile, by increasing the pH above pH 7.1 and towards pH 11, the surface charge was affected and decreased slowly between -25 mv and -29 mv towards -35 mv and -39 mv.

The particle size of SiO₂ aggregates is plotted as a function of pH in Figure 3b. At an initial pH of 7.1, the mean particle size of SiO₂ measures at 346 nm. When the pH was decreased, the particle size was found to increase rapidly from 4.0 to 2.8, with the highest mean particle size around 3250 nm. It is proven that aggregation occurred during pH 4.0 to pH 2.8. As the pH was decreased from 2.8 to 2.5, the particle size decreases rapidly to below 1500 nm. When the pH was increased from 7.1 to 11, the particles size remains below 500 nm. SiO₂ was in dissolution region when pH was higher was supported by the results obtained.

The particle size measurements in Figure 3b are concurrent with the surface charge values presented in Figure 3a. The mean particle size was shown to be the highest when in the range of pH_{PZC}. Furthermore, the particle sizes were also found out to be smaller when the surface charge was further from the point of zero charge. At PZC, the silanol groups (Si-OH) were stabilize due to the same amount of negative and positive charges of silanol groups created on the surface of the silica particles. The "hairy layer" in Figure 2 which consists of polysilicilic acid on the silica particle surface was also created in tap water at low pH with high ionic strength [11].

a)
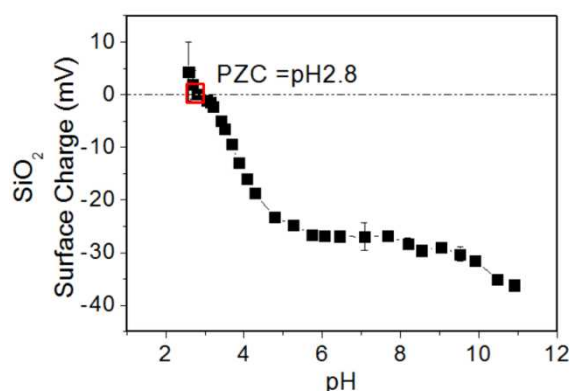
b)
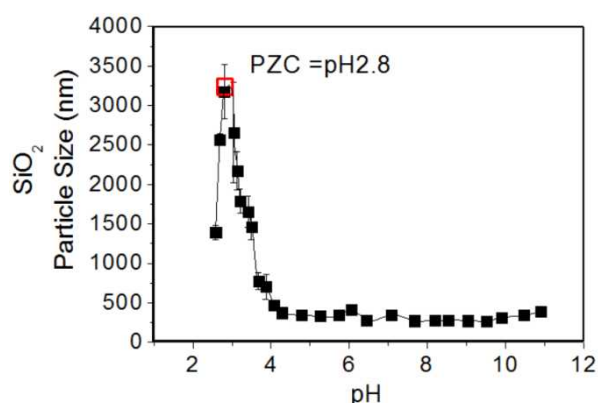
Figure 3(a) Surface charge of SiO₂ in tap water as a function of pH at a range of pH 2.5 to 11. The initial pH was found at 7.1 with a zeta potential of -27 ±2 mv. The pH of point of zero charge (PZC) was identified at 2.8. **(b)** Particle size

measurement of SiO₂ in tap water as a function of pH at a range of pH 2.5 to 11. The initial pH was found at 7.1 with a particle size of 346 ±24 nm. The pH of point of zero charge (PZC) was identified at 2.8.

Comparison of Characterization of SiO₂ nanoparticle in DI water and tap water

The first different of SiO₂ nanoparticle in DI water and tap water is the initial pH of tap water is neutral (7.1) and DI water is slightly acidic (5.4). It shows that tap water has other chemical ions which able to neutralize the pH since DI water are just consist of OH- and H+ ions besides consisting of SiO₂ nanoparticle. The second different of SiO₂ nanoparticle in DI water and tap water is tap water has lower pH_{PZC} than DI water and the third different of SiO₂ nanoparticle in DI water and tap water is the mean particle size at pH_{PZC} in DI water is larger than tap water by around 1750 nm. Both second and third difference between SiO₂ nanoparticle in DI water and tap water indicated that unknown substances in tap water interfered on aggregation. In order to further understand the interruption caused by the other present ions in tap water, testing is required to run to identify the ions in tap water. According to Table 1, these are the four most common anions (Cl^-, SO_4^{2-}, NO_3^- and F^-) and four most common cations (K^+, Ca^{2+}, Na^+ and Mg^{2+}) that can be found in tap water of Malaysia by using capillary ion analysis [17]. For cations (K^+ and Na^+) of the tap water, the most possible situation that will occur inside the tap water will be silanol groups act as cation exchanging groups and form chemical equation as followed [10]:

$$SiOH + Me^+ + OH^- = SiO^- Me^+ + H_2O \qquad (6)$$

As for divalent cations Ca^{2+} and Mg^{2+}, both alkaline earth metals act as catalyst on silicic acid polycondensation [5].

However, the present of other anions group in tap water could reduce the chemical reaction happen in equation 6 by 'stealing' the common cation groups in tap water and positive charge silanol groups which end up decreasing the size of particles formed and results in required lower pH to reached PZC. As for anions of the tap water, the silanol group react with chloride ion might form as equation below [1]:

$$SiOH + H^+ = SiOH_2^+ \qquad (7)$$
$$SiOH_2^+ + Cl^- = SiOH_2^+ \dots Cl^- \qquad (8)$$
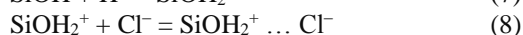
For F^-, the silanol group might form as equation below [9]:

$$SiOH + F^- = SiF + OH^- \qquad (9)$$

Meanwhile, it is proposed that anions such as Cl^-, SO_4^2 will affect aggregation formed by metal ions and silica, but not NO_3^- [6]. As a result, it not only reduced other present cations in tap water to react with silica, but also decreased silica self-aggregated.

TABLE 1. Concentration profiles of the common anions and cations in tap water sample in Penang, Malaysia [17].

Water sample	Anion				Cation			
	Cl^-	SO_4^{2-}	NO_3^-	F^-	K^+	Ca^{2+}	Na^+	Mg^{2+}
Tap water								
Day 1	5.2	8.0	1.3	1.9	2.5	8.2	3.3	3.7
Day 2	5.4	8.2	1.7	2.1	2.5	9.1	3.1	3.7
Day 3	5.5	8.4	1.7	2.2	2.6	8.5	3.2	3.8
Day 4	5.6	8.5	1.7	2.4	2.6	8.8	3.2	3.7
Day 5	5.5	8.4	1.7	2.4	2.6	8.7	3.2	3.7
Day 6	5.7	8.3	1.7	2.4	2.7	7.7	3.2	3.4
Day 7	5.8	8.0	1.8	2.4	2.5	8.1	3.2	3.5

Concentration (mg litre⁻¹) of mineral

CONCLUSION

Particle size and surface charge measurements were used to simultaneously investigate the interactive processes and stability of suspensions containing SiO₂ NPs in DI water and tap water. Our results clearly show that aggregation or disaggregation SiO₂ NPs is dependent on the variation of pH. Our study also indicates that SiO₂ NPs in tap water is much more difficult to treat than DI water by finding out the aggregation of SiO₂ NPs in tap water is 1750 nm smaller than DI water when both in the range of pH_{PZC}, which is the aggregation domain. Meanwhile, the potential interaction of SiO₂ NPs with DI water and tap water also had been studied. By having a better understanding of the characteristics of SiO₂ NPs, the performance of water treatment system can be improved by using more suitable coagulant which can be analysed using dynamic light scattering (DLS) method as well, thus benefit industry area in achieving economic and environmental sustainability. DLS method provides concrete data in terms of surface charge and the corresponding particle size which facilitates in determining the optimum pH range for aggregation to occur. This subsequently economizes the expenses on chemical usage and sludge disposal. The zetasizer testing can also save a lot of time because the analysis only takes minutes and the result is quite accurate and convincing. Thus, it is highly recommended to apply this technique for water treatment system.

Prohibition of Chemical Weapons (W/5334-2 and 304/PAWAM/6050364/l100).

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Persian Abstract

چکیده

ی اکسید سیلیکون (SiO2) در مقیاس نانو در طی دو دهه گذشته به عنوان مواد زائد در آب رودخانه شناخته شده است و اخیرا اثرات نامطلوب برای سلامت انسان و حیوان، اکوسیستم و سیستم تصفیه آب داشته است. حذف نانوذرات SiO2 از آب هنوز به دلیل حجم کوچک و تعاملات نامطلوب درون بدن، چالشی است. در این مطالعه، روش پراکنش نور پویایی (DLS) برای مشخص کردن SiO2 از لحاظ سطح بار و اندازه ذرات به عنوان تابع PH در محدوده ۲ تا ۱۱ به منظور تحلیل رفتارهای تجمع و اهمیت متقابلهای درون متقارن در (deionized) DI آب و آب شیرین. تجزیه و تحلیل DLS هر دو pH مقادیر نقطه صفر (PHPZC) NP های SiO2 را در آب DI و آب شیرین در pH 3.2 و pH 2.8 به ترتیب مشخص نمود. pH اولیه در دمای ۷٫۱ درجه سانتیگراد در آب آشامیدنی با میانگین اندازه ذرات ۳۴۶ نانومتر و مقدار متوسط سطح شارژ ۲۷- میلیمتر در مقایسه با pH اولیه آب DI که ۵٫۴ با میانگین اندازه ذرات ۲۹۵ نانومتر بود و سطح متوسط سطح ارزش ۳۳- متر مکعب مشخص شد که هر دو در آب آب شیرین و آب شیرین، SiO2 NPs جمع شده و افزایش در اندازه ذرات، اما کاهش در بار سطحی زمانی که PH به آرامی به سمت PHPZC مربوطه خود را از pH اولیه با اضافه کردن M۰٫۲۵ اسید هیدروکلریک کاهش می یابد. اندازه ذرات متوسط در pHPZC در آب DI در اندازه ۱۷۵۰ نانومتر در مقایسه با اندازه ذرات متوسط آب شیرین اندازه گیری می شود که نشان می دهد حضور یون های دیگر در آب شیرین فرآیند تجمع را مهار می کند. در نتیجه، نتایج نشان می دهد که pH بر سطح سطوح NP SiO2 تأثیر می گذارد و رفتار پایداری و فرایندهای متقابل آن را در حالت تعلیق های آبی تاثیر می گذارد.