



Preparation and characteristics of self-floating silica

Ghulam Muhyodin^a, Xiaoqun Zhu^a, Masroor Abro^b, Ghulam Yaseen^{a,c}, Inamullah Maitlo^a,
Muhammad Yasir Akaram^a, Jun Nie^{a,*}

^a State Key Laboratory of Chemical Resource Engineering, School of Materials Science and Engineering, Beijing University of Chemical Technology, Beijing, 100029, PR China

^b College of Chemical Engineering, Beijing University of Chemical Technology, Beijing 100029, PR China

^c College of Energy, Beijing University of Chemical Technology, Beijing 100029, PR China



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ABSTRACT

Tetraethyl ortho-silicate (TEOS) and octadecyl 2-((3-(triethoxysilyl) propyl) amino) acetate (SP) are used as modification agents at the ratios of 1:5, 1:1, 4:1 and 1:0 of TEOS: SP to form the silica by sol-gel process. Silica in both liquid form as well as powder forms at different compositions by weight percent have been subjected to the photo-polymerization with polyethylene glycol diacrylate (PEGDA) in the presence of photo initiator (2-hydroxyl - 2-methyl - 1-phenyl - 1-prop none (1173)). FTIR study is performed to identify various functional groups in the prepared materials, whereas, TEM and SEM are conducted to investigate particle size and surface morphology respectively. Moreover, transition glass temperature, storage and loss modulus are reported via DMA. The results indicated that TEOS: SP ratio has proved to be highly significant, among them 1:5 ratio is the most beneficial in terms of yielding maximum self-floating, hydrophobicity and roughness with minimum glossy nature of silica.

1. Introduction

Self-floating silica are considered as admirable material having desirable properties such as high surface roughness, high hydrophobicity and low glossy nature. By virtue of these properties, these kind of materials have a variety of practical applications to coat different surfaces such as ceramic, glass, metals, exterior parts of automobile, aircraft external body, wind turbine blades, space shuttles and ice-repellent etc [1]. Since these exhibit the high hydrophobicity, so these are regarded as self-cleaning materials. Based on their versatile nature, floating silica are of special interest from both academic and industrial point of view [2].

Increased hydrophobicity can be achieved either by roughening the materials having low surface energy or by modifying them chemically [3–5]. For this purpose, various techniques such as lithographic patterning [6], sol-gel technology [7–9], plasma etching [10], chemical vapor deposition [11], electrochemical deposition process [12–14] layer-layer assembly plus phase separation process [15] have been practically used. Among all these, sol-gel method is considered to be more advantageous and extensively used [16–21], since it is enriched with many characteristics features such as, high reliability, high efficiency, desirable uniformity and most importantly, large and complex shape substrates can be easily treated under the economical operating

conditions [3,14,22]. Many researchers have applied this method for preparing the high hydrophobic silica coatings through the hydrolysis and condensation of tetra methyl orthosilicate (TMOS), tetra ethyl orthosilicate (TEOS) [23,24]. Lakshmi et al. [6] combined a perfluoroalkylmethacrylic copolymer with a hybrid sol-gel matrix containing fumed silica nano-particles to get super hydrophobic sol-gel nanocomposite coatings. Nakajima et al. [17] prepared hard super hydrophobic thin films as a result of combining a phase separation of TEOS, induced by the addition of an acrylic polymer and subsequent fluoroalkylsilane coating. Latthe et al. [25] applied sol-gel method in acidic condition to prepare the water repellent Silica on glass substrates using two hydrophobic agents, namely, tetra ethyl orthosilicate (TEOS) and vinyl trimethoxysilane (VTMS), to increase the hydrophobicity they further used co-precursors. Purcar et al. [26] synthesized the hydrophobic and antireflective coating under acidic conditions by sol-gel method using tetraethylorthosilicate (TEOS) as a precursor and octyltriethoxysilane (OTES), phenyltriethoxysilane (PTES), methyltriethoxysilane (MTES) and vinyltriethoxysilane (VTES) and as surface modifiers. Petcu et al. [1] prepared the silica hybrid monolayer and bilayer coating through sol-gel method on propylene substrate. They used different silica pre-cursors for preparation of monolayer coatings to enhance the hydrophobicity of selected substrate. Furthermore, they added another coating with monolayer coating to prepare the bilayer

* Corresponding author.

E-mail address: niejun@mail.buct.edu.cn (J. Nie).

coating that resulted in the modification of its physicochemical properties. To produce super hydrophobic sol–gel nanocomposite coating, Basu et al. [7] used spraying precursor mixture containing hydrophobically modified silica nanoparticles dispersed in sol–gel matrices, which were further obtained via acid-catalyzed hydrolytic condensation of TEOS and MEOS. Gu et al. [27] synthesized transparent super hydrophobic thin films by dip-coating the silica sol on a glass slide, based on the self-assembly of silica nanoparticles prepared by hydrolyzing TEOS in the ethanol solution of NH_4OH and heptadecafluorodecyl trimethoxysilane.

In current study, the octadecyl 2-((3-(triethoxysilyl) propyl) amino) acetate (SP) was synthesized, which further was used along with TEOS at different mass ratio in the preparation of silica via sol-gel method. As a result, a long hydrocarbon chain was introduced onto the surface of silica under the adopted protocol, thereby, controlling the floating behavior of silica in solution. Silica was purified by three consecutive cycles of washing and centrifugation, followed by drying in an oven at 43°C , which resulted in the formation of powdered silica. Afterward, the floating silica both in liquid and powder forms were subjected to undergo the photo-polymerization in polyethylene glycol diacrylate (PEGDA) using a photo initiator (2-hydroxyl – 2-methyl – 1-phenyl – 1-propanone (1173)) on a glass substrate. This study is attempted with the purpose of achieving the floating behavior of silica, decreasing its use and increasing the overall surface properties such as hydrophobicity, surface hardness and decrease the glossy nature. Also this process is convenient to carry out at lab with easy handling.

2. Materials and experimental

2.1. Materials

Octadecyl acrylate (ODA) and 3-amino propyl triethoxy silane (KH550) were purchased from Beijing Lark Technology Co. Ltd and Shanghai Macklin Biochemical Co. Ltd. Tetra ethyl orthosilicate (TEOS), hexanediol diacrylate (HDDA) were purchased from Sigma Aldrich Co. Ltd and Tianjin Long Days Co. Ltd. Polyethylene glycol diacrylate (PEGDA), 2-hydroxyl – 2-methyl – 1-phenyl – 1-propanone (1173) were purchased from Aladdin industrial corporation and Tianjin Jiuri Chemical Co. Ltd. (Tianjin, China). Ammonia ($\text{NH}_3\text{H}_2\text{O}$ – 25% A.R) and ethanol were purchased from Beijing chemical factory.

2.2. Synthesis of octadecyl 2-((3-(triethoxysilyl) propyl) amino) acetate (SP)

SP was prepared by carrying out a reaction between octadecyl acrylate and 3-aminopropyl triethoxysilane. On the basis of their 1:1 molar ratio, 15 g of octadecyl acrylate (ODA) was poured in the 3-necked flask and 10.23 g of 3-aminopropyl triethoxysilane (KH550) was added in drop-wise manner at approximate flow rate of 0.85 mL/min. The flask was immersed in the water bath placed on the bidirectional magnetic stirrer. Mixture was continuously stirred for 6 h at the temperature of 45°C in the presence of nitrogen gas purge. Chemistry involved in this synthesis is depicted in Scheme 1(a).

2.3. Preparation of the silica particles

In the first step, tetra ethyl orthosilicate (TEOS) and SP were mixed with each other with different mass ratio of 1:5, 1:1, 4:1 and 1:0 separately. In addition to these, optimized amount of other ingredients including H_2O (2 mL), $\text{NH}_3\text{H}_2\text{O}$ (25% A.R) (0.1 mL) and ethanol (1 mL) were also added, which altogether, formed a silica through hydrolysis and condensation reactions within 1.5 h. Then, 5 g of HDDA was added drop-wise within 45 mins. The mixture was continuously and drastically stirred under the ambient temperature. Table 1 presents the details of silica samples liquid (S1, S2, S3 and S4) and solid (S5, S6, S7 and S8) mass ratios of TEOS: SP respectively. Scheme 1(b) gives the

representation of synthesized silica process. The centrifugation was applied thrice to silica at 8000 RPM for 15 min, to wash away water, $\text{NH}_3\text{H}_2\text{O}$ and HDDA using approximately 20 mL of ethanol. The powdered silica was yielded. The sample was placed in vacuum oven at 43°C for 6 h.

2.4. Photo-polymerization

Different weight percent of liquid and powdered silica were added in polyethylene glycol diacrylate (PEGDA) in the presence of 3% photo initiator (1173). It was deposited on the glass substrate and exposed to UV-365 at light intensity of 22 mW/cm^2 for 60s. The detailed photo-polymerization formulation is provided in Table 2. Schematic representation of photo-polymerization is shown in Scheme 1(c).

2.5. Characterizations

The average particles size of silica was measured by using the TEM (Techai G2 T20, FEI, USA) at ambient temperature. The silica dispersion was diluted with (0.3 mg in 25 mL ethanol) and homogenized with an ultrasound bath (2 h) at 90°C . The wetting properties of the silica deposited onto the glass substrate were studied in terms of water contact angle (θ) using deionized water droplet on JC 2000 (voltage 220 v, frequency 50 Hz,) equipped with a digital camera at ambient temperature. The hardness was measured by using the rocker hardness tester QBYII according to standard value of glass $440 \pm 6\text{ s}$ and should not exceed the value of 1 to meet the level of satisfaction. Glossy Meter WGG60-E4 was used to measure the glossy nature of polymer samples. Reported parameters were measured as mean of three values obtained on different locations. The surface morphology was studied by Scanning Electron microscopy (SEM ZEISS Supra55, EHT 20KV). EDX oxford instruments LNCA x-act was used to analyze the silicon, carbon and oxygen. The deformation of polymer was reported by using DMA (NETZSCH 242C, Germany) with provided conditions of sample such as length of 3 cm, width of 6 cm and height of 2 mm at a heating rate of $5^\circ\text{C}/\text{min}$. FT-IR was studied in the range of $600\text{--}4000\text{ cm}^{-1}$ by using thermo scientific IDB (ATR) spectrometer with a resolution of 4 cm^{-1} .

3. Results and discussions

3.1. FT-IR

The preparation of SP was verified by FT-IR spectra obtained in the range of $2000\text{--}750\text{ cm}^{-1}$, which is given in Fig. 1(a). The peaks in the range of $1680\text{ cm}^{-1}\text{--}1640\text{ cm}^{-1}$ prove the existence of C=C double bond in acrylate. During the reaction, double bond was disappeared, forming a new C–C single bond and C–N bond between nitrogen of 3-amino propyltriethoxysilane and carbon of octadecyl acrylate. The change can be visualized in the form of peak ranging between 1600 and 1585 cm^{-1} . Variation in the peaks before and after Micheal Addition Reaction confirms the formation of new bonds.

The IR-ATR spectrum of silica is reported in Fig. 1(b). TEOS, prior to reaction with SP, it has undergone the hydrolysis and alcohol condensation, as a result of which a symmetric Si–O–Si network in the hybrid film was formed, as it is confirmed by the peak obtained in the range of $1100\text{--}1000\text{ cm}^{-1}$. Previously, it has been reported by the Pectu et al. [1], that, this symmetric network moves to the higher wave-number of 1043 cm^{-1} resulting the asymmetric stretching vibration caused by presence of C–Si bond from organic function. This causes the Si–O–Si symmetry linkages from the hydrophobic Silica (hybrid) network to be destroyed [28]. The spectrum also shows the existence of CH_2 - and CH_3 functional groups by the peaks obtained at 2855 cm^{-1} and 2932 cm^{-1} respectively. These spectra proved the existence of all the expected bonds like C–N, C–Si, C–O, Si–O–Si, $-\text{CH}_2$ and $-\text{CH}_3$.

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