ZEWAIL CITY: UNIVERSITY OF SCIENCE AND TECHNOLOGY

Experiment (I) - Silica Microparticles

Introduction to Nanomaterials Synthesis (MATSCI 255) – Spring 2020 – Instructor: Prof. Mohamed Bassyouni – Performed: 6/2/2020 – Submitted 13/2/2020

Students: Rami Wail – Ahmed Khaled – Ahmed Alaa – Mennatallah Khaled ID: 201600112 – 201601309 - 201600352 - 201601533

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I. INTRODUCTION AND PURPOSE

The objective of this experiment is the synthesis of and characterization of silica micro/nanoparticles and review of the applications of microporous materials in various industries. Firstly, silica, or silicon dioxide (SiO_2), is a group IV metal oxide, which naturally occurs in both crystalline and amorphous forms [1]. The various forms of crystalline silica are: α-quartz, β-quartz, α-tridymite, β-tridymite, α-cristobalite, β-cristobalite, keatite, coesite, stishovite, and moganite [1]. The most abundant form of silica is α-quartz, which is mostly known as crystalline silica [1]. It is worth mentioning, that silica nanoparticles exhibit unique characteristics at the nanoscale due to their porous morphologies. Accordingly, these nanometric pores can be utilized in an extensive range of practical applications in various industrial and commercial fields. Nanoporous materials exhibit diameters less than 100 nm, however, are further classified into three bands based on pore size as follows with varying transport mechanisms:

- Microporous, having pore size smaller than 2 nm which is comparable to that of molecules. Activated transport dominates.
- Mesoporous, between 2 and 50 nm, same order or smaller than the mean free path length. Knudsen diffusion and surface diffusion occur. Multilayer adsorption and capillary condensation contribute.
- Macroporous, larger than 50 nm, larger than the typical mean free path length of typical fluid. Bulk diffusion and viscous flow.

On the other hand, the production of uniform silica microspheres with adjustable pore size has proven to be an unfortunate challenge for nanotechnologies engineers. However, silica achieves physical and chemical properties that make it suitable for many industrial and commercial uses. The three predominant commercial silica product categories are: sand and gravel, quartz crystals, and diatomites. Conclusively, some of the various industries that utilize silica are introduced in Table 1: -

Table 1: Industrial uses of silicon [1]

Glassmaking (Sand)	Containers, flat (plate and window), speciality, fibreglass (un-ground or ground)
Foundry	Moulding and core, moulding and core facing (ground), refractory
Metallurgical	Silicon carbide, flux for metal smelting
Abrasives	Blasting, scouring cleansers (ground), sawing and sanding, chemicals (ground and un-ground)
Fillers	Rubber, paints, putty, whole grain fillers/building products
Ceramic	Pottery, brick, tile
Filtration	Water (municipal, county, local), swimming pool, others
Petroleum industry	Hydraulic fracturing, well packing and cementing
Recreational	Golf course, baseball, volleyball, play sands, beaches, traction (engine), roofing granules and fillers, other (ground silica or whole grain)
Gravel	Silicon, ferrosilicon, filtration, non-metallurgical flux, other
Electrical	transistors, MOSFETS, LED's etc.

It is worth mentioning that silica minerals compose approximately 26% of Earth's crust by weight [2]. Whereas silica as a compound in the form of silicon dioxide, constitutes 59% of Earth's crust [3]. Finally, it is to be noted that silica exhibits different molecular structures depending on the pH of the media. According to the Stöber process proposed by Werner Stöber and his team in 1968, particle size and distribution vary with pH as shown in Figure 1: -

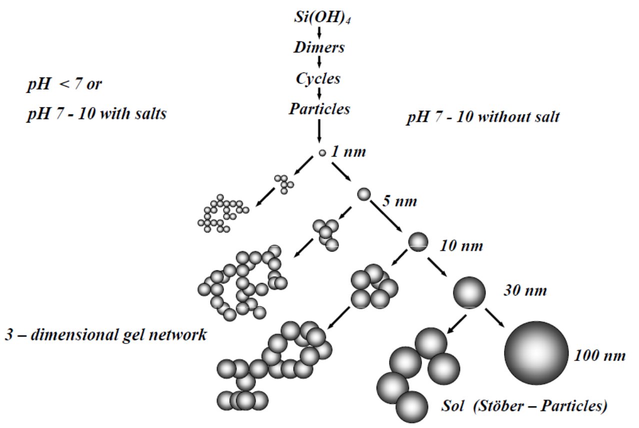

Figure 1. Morphology of silica nanoparticles (Brinker & Scherer)

It can be concluded that single particles are found in chemical solutions whereas cycles of particles form a dimensional gel network when in basic solutions.

II. Materials

The used chemicals included:

- Ethanol of known concentration
- Distilled water
- Tetraethyl orthosilicate (TEOS)
- Ammonia

The used tools included:

- Two closed bottles
- A graduated cylinder to quantify used ethanol
- Pipettes to quantify ammonia, distilled water and TEOS
- Magnetic stirrer and heater
- Centrifuge

III. Method and Procedure

The final target of the procedures is to obtain silica nanoparticles of two sizes (100 nm and 500 nm). Two samples are prepared in two bottles. Firstly, 50 mm^3 of ethanol is transferred to each bottle. Secondly, 1 mm^3 of distilled water is added to each sample. Thirdly, 2.7 mm^3 of ammonia is put added to bottle 1 and 5.4 mm^3 of ammonia to bottle 2. Then, both samples undergo magnetic stirring and are subjected to heating at 60 degrees Celsius for five minutes. Then, 2.5 mm^3 of TEOS is added to both samples. They are left in the same conditions for an hour. Finally, the samples are put into the centrifuge at 6000 rotation per minute for a couple of minutes.

Figure 2: After adding ethanol, distilled water and ammonia, the two samples are put on stirrer at 300 rpm and 60 degrees Celsius.

Figure 3: Adding TEOS to the sample after stirring for five minutes.

IV. Results

1) After about 30 minutes of warm stirring (after adding the TEOS) at 60 degrees Celsius and 300 rpm, the solution in bottle 2 was noticed to be highly turbid while that in bottle 1 showed a much less turbidity. See figure 4.

Figure 4: 30 min

2) Then after about 90 minutes from adding the TEOS, the degree of turbidity of both bottles was highly increased as shown in figure 5.


Figure 5: 90 min

After the Centrifuge, we could see the fine powder of the nano silica as a white precipitate.

V. Discussion and analysis

- Since TEOS is the source of silica, and since we added the same amount of silica in both bottles, then we should expect the same yield from both bottles although each will result in a different size particle.
- The size of the particles depends on the conditions of the process, i.e., the pH of the medium. The determination of the steps and conditions that are required to form particles with a certain size is determined experimentally.
- Ammonia is used in this process as a catalyst. Thus, in bottle 1 with the higher amount of ammonia, the particles are formed faster than bottle 2 as indicated by the difference in turbidity in both bottles.

VI. Concllusion

The goal of the experiment was to prepare two different sizes of silica nanoparticles 100nm in the first bottle and 500 nm in the second bottle. The procedure was the same for both samples adding ethanol, then distilled water, ammonia finally Tetraethyl orthosilicate (TEOS). The concentrations were the same for both samples except ammonia was 2.7 mm^3 for the first bottle and 5.4 mm^3 for the second bottle.

Then the samples undergo string alongside heating. Second bottle showed turbidity at a higher rate than the first bottle. Both samples showed turbidity clearly after 90 minutes. Finally, both samples undergo centrifugation to precipitate the white ppt from the solution then they were dried. The higher amount of ammonia in the second bottle caused reaction rate to be higher than that of the first bottle which caused the difference in size for both samples as expected.

References

[1] Humans, I. (2012). Arsenic, Metals, Fibres and Dusts. International Agency for Research on Cancer.
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[3] Encyclopedia Britannica. (2019). silica | Definition & Facts. [online] Available at: https://www.britannica.com/science/silica [Accessed 12 Feb. 2020].