EXTRACTION OF SILICON NANOPARTICLES FROM RIVER SAND IN A MECHANO-CHEMICAL APPROACH

Md. Rounakul Islam and Abu Bakar Md. Ismail

***Abstract*—** Extraction of silicon (Si) from river sand and separation of silicon nanoparticles (Si-NPs) from the extracted silicon has been investigated in this report. Thermite process was used to reduce silicon from sand and in this process Si was extracted as Si-Al alloy, which was further refined by acid leaching. Then it was heated and dry Si powder was found which might be in oxidized form. To remove oxidation, etching process was used by mixing 2-propanonol and 48% HF acid with a specific ratio and the etching process was terminated by adding excess 2-propanol. The obtained Si powder was tested XRD (X-ray diffraction), and XRF (X-ray fluorescence) to study the properties of the obtained Si. Then sonication was performed to de-agglomerate and disperse silicon particles into the 2-propanol solvent. The main focus of this experiment was to vary the sonication time and investigate variation in the mean diameter of the particles which was confirmed by using DLS (Dynamic Light Scattering) technique accordingly. Si-NPs of mean diameter of 92.7 nm was found after 6 hours of sonication. Solution found from different sonication time was tested with UV-Vis spectrometer to study the optical characteristics of Si particle found after sonication.

***Keywords*—** Thermite, Etching, Sonication.

I. INTRODUCTION

SILICON is one of the most abundant (27.7% of the earth's crust) elements, which is easily extracted from white sand (SiO_2) in an exothermic process [1]. Typically, silicon (Si) makes up the majority of the elements that make up sand [2]. It is very brittle and a metalloid with a split metallic cluster [3]. Flint, quartz, and opal are just a few of the various forms of silicon that may be found in nature. In actuality, roughly 35 different crystalline forms have been found [4]. Traditionally, silica is extracted from rock using a carbothermic method that involves heating charcoal at temperatures between 1500 and 2000 °C [5]. Over 95% pure Si is produced by this carbothermic method [6]. However, this technique consumes a lot of energy and also liquefies the silicon, erasing any original SiO_2 form. Additionally, single crystal silicon is not feasible, it is highly expensive, and the furnace must operate at temperatures exceeding 2000 °C [7-8]. The cost of the production of silicon is quite high and it is an irreplaceable raw material in the electronics industry, so several methods are currently being under research to make semiconductor devices. There are several alternative ways to extract silicon from sand there, but the alumino-thermic technique appears to be the least expensive and environmentally friendly method as well [9-10]. Contrarily, because it may be carried out at a reasonable temperature, the aluminothermic reaction is regarded as one of the simplest methods of collecting silicon from sand [11-12]. To remove the extra components from the reaction products, common purification methods such electrolysis, acid treatment, or fractional crystallization are applied.

A few mechanisms are there to produce Si-NPs such as laser ablation [13], chemical vapor deposition (CVD) [14], sol-gel method [15], ball milling [16] and electrochemical etching [17]. Laser ablation is a fast and simple method for producing silicon nanoparticles, but it requires expensive equipment and can only produce small quantities of nanoparticles at a time. Additionally, the nanoparticles produced by laser ablation can be contaminated with impurities from the target material or the surrounding environment. CVD is a widely used technique for producing silicon nanoparticles, but it can be expensive and complex to set up. The process requires high temperatures and specialized equipment, and the nanoparticles produced can be highly reactive and prone to oxidation. The sol-gel method is a relatively simple and cost-effective way to produce silicon nanoparticles,

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but it can be difficult to control the size and shape of the particles. Additionally, the nanoparticles produced can be prone to agglomeration, which can affect their properties and performance. Ball milling is a mechanical method for producing silicon nanoparticles that is relatively low-cost and easy to implement, but it can result in the formation of large aggregates or clusters of nanoparticles. Additionally, the grinding process can introduce impurities or defects into the nanoparticles, which can affect their properties. Electrochemical etching is a simple and inexpensive technique for producing silicon nanoparticles, but it requires specialized equipment and can be difficult to control the size and shape of the particles. Additionally, the nanoparticles produced can be prone to oxidation or contamination from the electrolytic solution. One of the simplest methods for manufacturing silicon nanoparticles (Si-NPs) from this purified silicon is the mechano-chemical process [18]. This article describes the manufacturing of Si-NPs first from river sand combining an alumino-thermal technique and a mechanical approach that produces no CO_2 at moderate temperatures and low expense.

II. EXPERIMENTAL METHODOLOGY

A. Extraction of Silicon from River Sand

Sand is the raw material and it was collected from the bank of Padma River in Rajshahi, Bangladesh. To get rid of chemical contaminants, the collected sand was treated with regular water (5 times), purified water (3 times), and finally acetone (3 times). The washed sand was dried in a furnace (Carbolite CWF) at 100 °C for roughly two hours. A mortar and pestle was used to pound dry sand into small particles. Sand was ground into an extremely tiny particle since the reaction becomes faster if the sand is finer. Si powder was uniformly combined with aluminum (Al) and sulfur (S) in a clay pot at a particular weight ratio of 8:4:7. To hasten the thermite reaction, a very little degree of magnesium (Mg) was also added to the mixture. By lighting the mixing bowl with a magnesium ribbon, the reaction was sparked outside. The reaction took place for around 30 seconds, after which the end product was left to naturally cool. After that, silicon was separated as Si-Al eutectic compounds, which were then three times refined by acid leaching to get pure silicon. To slightly cleanse the additional components and contaminants in the extracted Si, a leaching procedure was carried out using 10 ml HCl (48%) in a glass beaker for 4 g of Si-Al ground mixture at 60 °C for 15 min. This procedure was done three times. The customized alumino-thermic process is depicted in the equations below along with its complete process, which is represented in Figure 1.

$$2Mg + O_2 \rightarrow 2MgO + \text{energy}$$

$$2Al + 3S \rightarrow Al_2S_3 + \text{energy}$$

$$3SiO_2 + 4Al \rightarrow 2Al_2O_3 + 3Si + \text{energy}$$

$$Al_2S_3 + 6HCl \rightarrow 2AlCl_3 + 3H_2S$$

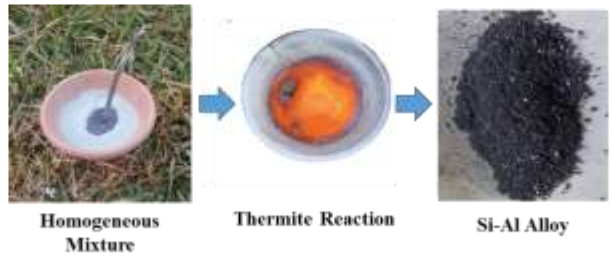

Figure 1: Aluminothermic Silicon Extraction Process.

B. Synthesis of Silicon Nanoparticles: Mechano-chemical Approach

Due to its better stability, 2-propanol was chosen as the solvent for the silicon particles. In order to dissolve the particles of silicon into 2-propanol solvent, a chemico-mechanical procedure was adopted. Ultrasonic vibrator was used to disperse and de-agglomerate silicon particles into liquids. High shear forces produced by ultrasonic cavitation separate particle agglomerates into individual particles. The dispersed solutions were divided into three groups based on the duration of the sonification, which was done for 120 min, 240 min, and 360min. The sedimentation method was used to separate the particles. By changing the sonification time, the sedimentation time was kept constant to separate the particles. The size distribution profile of silicon particles was ascertained using the DLS technique on these separated particles dispersed in 2-propanol.

Figure 2: Mechano-chemical approach to synthesize Si-NPs.

III. EXPERIMENTAL RESULTS AND DISCUSSION

A. X-ray fluorescence (XRF) Analysis

In a meticulous X-ray Fluorescence (XRF) analysis of extracted silicon, as depicted in Figure 3, shows the purity of silicon over 97%. As the XRF spectrometer unveils a pristine elemental profile dominated by silicon, with a mere trace of manganese, an almost elusive guest in this composition. What's striking, however, is the absence of any other contaminants. This silicon sample stands as a testament to the meticulous care taken to ensure its exceptional purity.

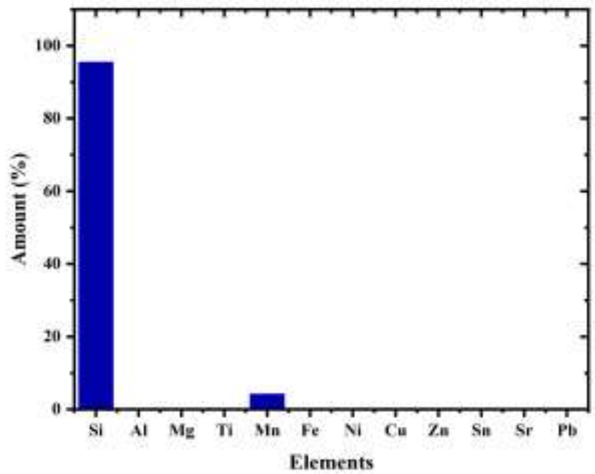

Figure 3: XRF report of the obtained silicon that was isolated following three acid leaching steps.

B. X-ray diffraction (XRD) Analysis

The presence of silicon was identified through phase analysis using the XRD technique. If the diffraction peaks are pushed on toward a lower diffraction angle, the crystal lattice is extended and impurity-related stress is minimized. Conversely, if the peaks are pushed into a higher diffraction angle, the crystal lattice is compressed. The XRD peak shift is caused by a move in the lattice properties as a result of several factors, including crystalline size and lattice strain, thermal annealing, doping-induced changes in stoichiometric composition, Doppler Effect during counts, etc. The polycrystalline silicon is visible in the XRD patterns of silicon that has undergone acid treatment.

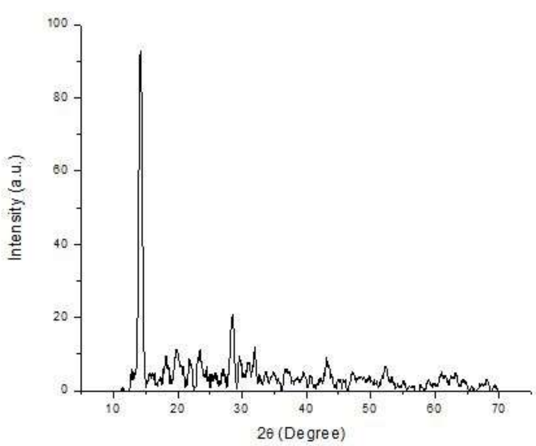

Figure 4: XRD (intensity vs 2Θ) of extracted Si obtained from aluminothermic reaction.

C. Particle Size Analysis with DLS Technique

Dynamic Light Scattering (DLS) was used to determine the particle size distribution profile which provides mean diameter of particle size. Sedimentation time was kept constant at 60 min, while only the sonication time was varied. Figure 5 shows the particle size distribution which was obtained by DLS technique. Since only sedimentation process was applied to separate the particles, a wide range of particle size was found. From Figure, it is shown that 120 min sonicated solution contains the Si particles of the mean diameter of 208.6 nm, 240 min sonicated solution contains the Si particles of the mean diameter of 144.2 nm and 360 min sonicated solution contains the Si particles of the mean diameter of 92.7 nm.

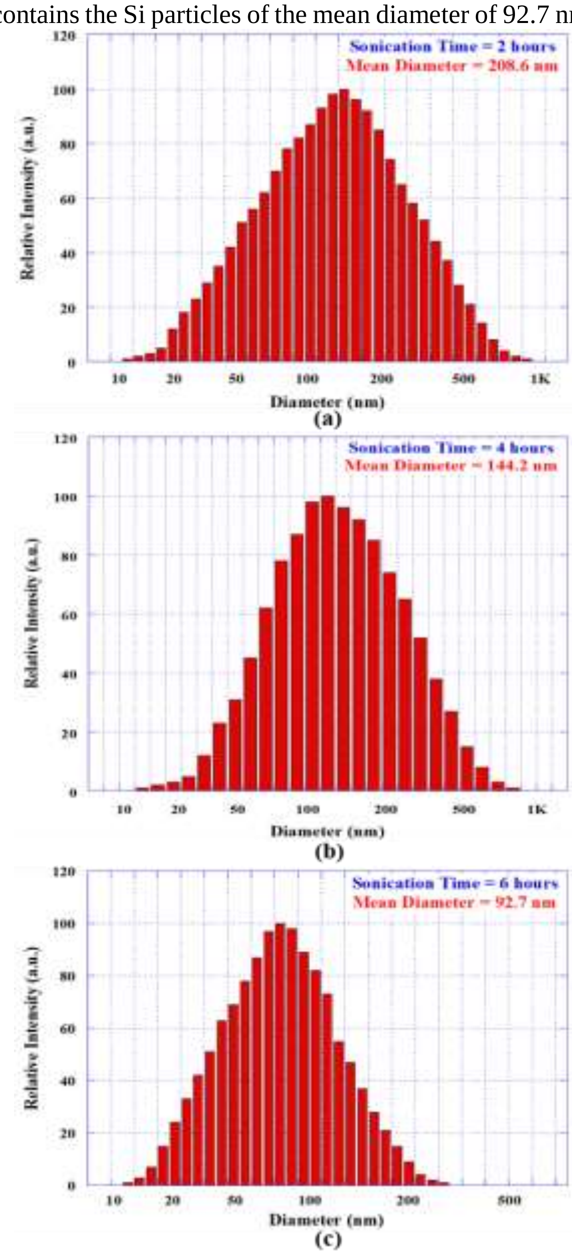

Figure 5: DLS report for (a) 120 min (b) 240 min and (c) 360 min sonicated solution.

D. Optical Characteristics

Solutions separated by means of different sonication times were used to perform UV-Vis absorbance test. It is observed that the absorbance of solutions increases with the increase in sonication time i.e. with the decrease in particle size.

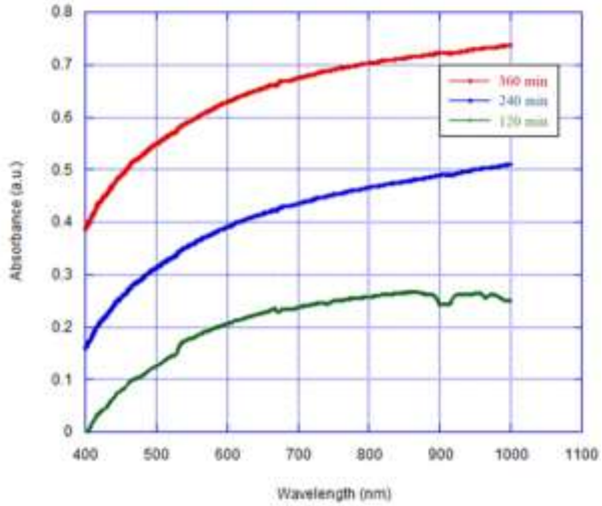

Figure 6: Variation in absorbance of solutions by varying sonication time.

IV. Conclusion

Silicon particles obtained from the Alumino-thermic reaction was processed through chemical leaching and etching to purify silicon particles which possesses more than 97% pure silicon. The extracted silicon was grinded and sonicated to fragment and disperse the silicon particles into the solvent 2-propanol. DLS report confirms the variation in mean diameter of the separated silicon particles with the variation in sonication time. From our experimental reports, it is obvious that the particle size less than hundred nanometer is possible to separate by following this mechano-chemical approach with low cost, highly purified and in an environment friendly way. It can be concluded that the mean diameter of silicon particles can be reduced to a few nanometer with the increase in sonication time

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