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ABSTRACT

In this research, we have investigated the extraction of silicon from sand by Metal-thermite reduction process. The sand was collected from Padma River near Rajshahi city, Bangladesh. The reduction of sand was performed with Al and Mg powder. After that leaching out with HCl (Hydrochloric acid) for purification. We have studied the structural, chemical nature and physical morphology of the extracted sample. Using X-ray Diffraction (XRD) measurements, the structural analysis of the samples was taken and confirmed that the element was produced Si which has the particle size in an average 31.52 nm. In SEM (Scanning Electron Microscopy) was used for surface morphology of obtained samples. FTIR (Fourier Transform Infrared Spectroscopy) was provided the presence of elemental percentages of Silicon. XRF (X-ray Fluorescence) was used for characterization the chemical nature of sand. Silicon content of sand of Padma River was found about maximum 55% in this study of Al thermite reduction process. From Optical analysis it had found the band gap is1.249 eV.

Keywords: sand, reduction, leaching, combustion etc.

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I. INTRODUCTION

Silicon is a prominent and vital element to many human industries for electronic and biological devices likely manufacturing computer chips, transistors and integrated circuits [1, 2]. It has extensively used in optoelectronics and solar cell materials. It has been involved the exploration of solar energy. Silicon (Si) is a metalloid element and the second most plentiful electropositive and the seventh most abundant (about 27.7%) element in the earth's crust other than oxygen. Silicon has specific gravity 2.42 gm/cm^3, but silicon has very rarely found as the pure element in the Earth's crust. Bangladesh is river best country which contains total length of river 24140 Km. In this river contain huge amount of sand. We get this sand easily and low cost process from that river. We can extract silicon from the sand various method. By this method we can extract silicon with low cost and this silicon can be used to fabricate solar cell. This solar cell we may use to produce huge amount of electricity without any hazard or environmental effect. This electricity may contribute in our socio economic. Due to this reason we extract silicon from sand. If we succeed the better amount (%) of silicon extract from the sand of river we may help to developed solar selective surface or plane that may be used in solar cell. In 1908 Hans Goldschmidt was first originated in thermite reduction process [3]. There were some well-known mechanism of thermite reduction process namely- Carbothermite, Calciothermite, Aluminothermite and Magnesium Thermite for reduction of Silica sand.

Industrially Carbothermic reduction process has been exerted for making silicon from sand which utilized electric arc furnace operating at greater than 2000°C [4,5]. This process was environmentally detrimental. Because Carbon

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monoxide was produced in Carbothermic reduction process [6]. Alumino Thermite process is environmentally benign process for extraction of silicon from sand [7]. We have used Aluminothermite reduction process for Silicon extraction by the reduction of sand with Al which is produced Silicon alloy. After that elemental Silicon is separated by leaching out with Hydrochloric acid (HCl). Silicon was Produced pure form. Due to acid leaching process dissolved the Al content in mineral Acid. The following reaction was held during reduction [8]-

$$2Al + 3S \rightarrow Al_2S_3 + energy$$

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

The important consideration is reducing tendency and the physical properties of the metal and the physical proper ties of its corresponding oxide in the selection of a suitable thermite system. If the thermite reaction is once initiated, it is capable of self-propagation because the large heat is released in this reaction. By assisting an energy source such as a combustion wave from a chemical reaction, electrical current, radiation energy, or a mechanical impact the reactions can be ignited. The initiation of the combustion reaction was affected by the physical and chemical stability of the reactant oxides. The combustion rate and combustion mode of thermite systems are affected by several factors. Due to the low cost of the reactant materials and the energy efficiency of the reactions, various material processes, for example, forming a ceramic lining in a pipe and storage of nuclear waste, have been manifested based on thermite reactions [9].

II. EXPERIMENTAL PROGRAM

2.1 Materilas

Sand, Magnesium powder, Aluminum powder, powdered sulfur, charcoal powder and HCl.

2.2 Procedure for sample preparation

The sample was prepared by following steps:

1. The Sand were collected from Padma River, washed by water 15 times and by distilled water 5 times. Then washed by acetone 5 times.
2. After washing dried at 120° C about 6 hours.
3. Then grinding the sand to form finer powder.
4. Homogenous mixture was made of Sand: Sulfur powder and Aluminum powder.
5. Mixing powder was poured to clay pot making cone shaped indentation at top ¾” deep filled with magnesium for facilitation ignition.
6. Using magnesium ribbon as fuse wire.
7. Waited about 20 minutes to cool the clay pot.
8. The pot broken apart and separated residue (Si-Al alloy) which were broken into small pieces using light tape hammer.
9. Then making fine power by mortar-pestle.
10. HCl was added with Si-Al alloy Powder in a glass beaker at 50-60°C on a hot plate for about 20 min.
11. Then additional HCl was added for further purification.
12. The silicon was found in the form of pea-size, hard, black, globules.
13. After drying prepare for characterization.

Fig.1. was shown the silicon extracted from sand.

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Fig.1: Extracted silicon from sand after leaching out

III. RESULT AND DISCUSSION

3.1 XRD Analysis

The XRD pattern of sample was shown in fig.2. which was produced by the reduction of sand particles with Aluminum and magnesium powder. The XRD peaks were recorded in the wide angle of

2θ from 20° to 50° with Cu kα (α=1.5406°) radiation. According to the figure the peaks appeared at 2θ range of 21.642°, 22.459°, 29.929°, 32.741°, 33.934°, 35.067°, 47.288°, 49.287° corresponds to the crystal planes of SiO_2 (220), SiO_2 (120), SiO_2 (400), SiO_2 (411), Si (200), SiO_2 (420), Si (220), SiO_2 (620) respectively. All peak positions are compared with the standard value according to the ICSD Collection Code: 67788 of Silicon. It is observed that two sharp peaks 33.907° and 47.256° indicate the presence of silicon crystal plan (hkl) values (200), (220) and some peaks are silicon dioxide (SiO_2),and remains are Al_2O_3 and Magnesium silicon compound $Mg(SiO_3)$ as impurity. All the peak are closely to standard values. By comparing these figure it is confirmed that obtaining XRD has silicon and small amount of impurity after leaching.

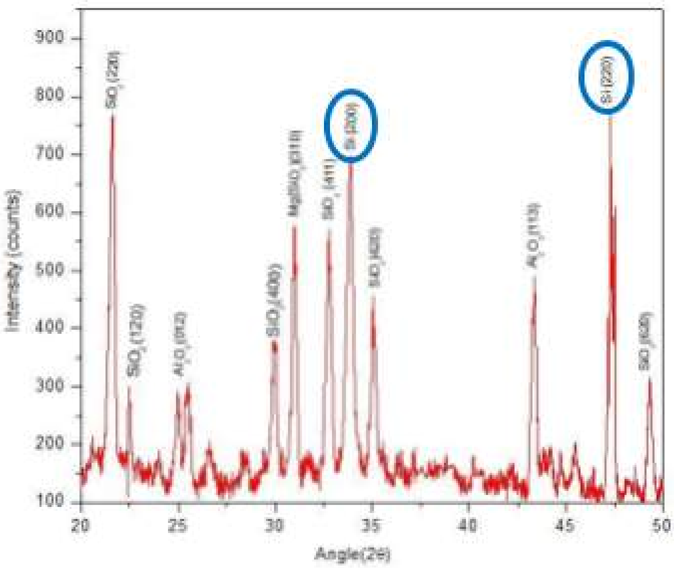

Fig.2: XRD spectra of Sample

From XRD study, the average particle size D has been estimated by following Debey-Scherrer equation, the average crystallite sizes are calculated:

$$D=K\lambda/\beta Cos\theta \quad (1)$$

Where K is the shape factor (the typically value is 0.94), λ (1.5406 Å) is the wave length of incident beam, β is the broadening of the diffraction line measured in radian at half of its maximum intensity (FWHM) and θ is the Bragg's angle and D is the diameter of the crystallite size. From Table I, The average crystal size of sample is 31.52 nm.

Table I: Measurement of crystallite size of sample

Observed peak position, 2θ (deg.)	Standard peak position, 2θ (deg.)	Observed relative Intensity, (%)	FWHM (degree)	Miller indices (hkl)	Crystallite size,(nm)
33.907	33.204	65%	0.445	200	18.86
47.256	47.666	96%	0.192	220	44.17

3.2 SEM Analysis

Scanning Electron Microscopy (SEM) has been used to observe the surface morphology of Silicon sample produced by Alumino-thermite reduction process. A general distribution of silicon particles in magnification X 19000 are shown in Fig. 3. It was observed that most of the particles are agglomerated but there some particles are identical which were shown in circle marked region. According to observations these particles size was around 25 to 40 nm. It has been comparable to the getting result from XRD data.

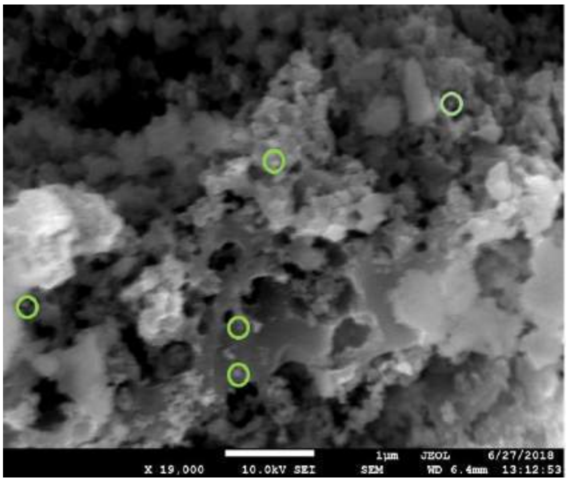

Fig. 3: SEM micrograph of Sample magnification X 19,000.

3.3 Results of FTIR

Fig.4. was shown the FTIR spectrum of silicon sample. FTIR spectra of obtaining samples were recorded 250 cm^{-1} to 4000 cm^{-1} by spectrum 100 Perkin Elmer in the central science laboratory, University of Rajshahi, Bangladesh. Respective vibrational modes for different functional groups were analyzed with standard FTIR data. The main peak at 471 cm^{-1} may be ascribed to the Si-Si Phonon mode as suggested by van de Walle and Lin. The peak 745 cm^{-1} is appeared as Al-OH bend. The wavenumber 1083cm^{-1} is for Si-O. Standard Peak at 1647cm^{-1} wavenumber is for Si-D stretching. The wavenumber 1644.53 cm^{-1} may be ascribed as Si-D. The wavenumber 3155 cm^{-1} is for O-H stretching bond. All peaks were closely to standard value of FTIR. From FTIR, it is assured that Si is present in the sample and some sort of Si-O bonding is also present. Because the reduction process is occurred properly.

3.4 XRF Analysis

XRF (X-ray fluorescence) was used for characterization of the chemical nature of sample. It is a commonly used technique for identification and qualification of elements in a sample. From XRF study, it is found that the prepared sample contains 54.1748% silicon. Others element like Mg (25.94%), Al (14%) and other minor element is present. . Except Mg and Al all other impurities from sand. Al and Mg impurity for externally supplied. It may have possible to get better purity of silicon by further leaching. Table II was shown the quantitative result of sample produced by the Aluminothermite reduction process.

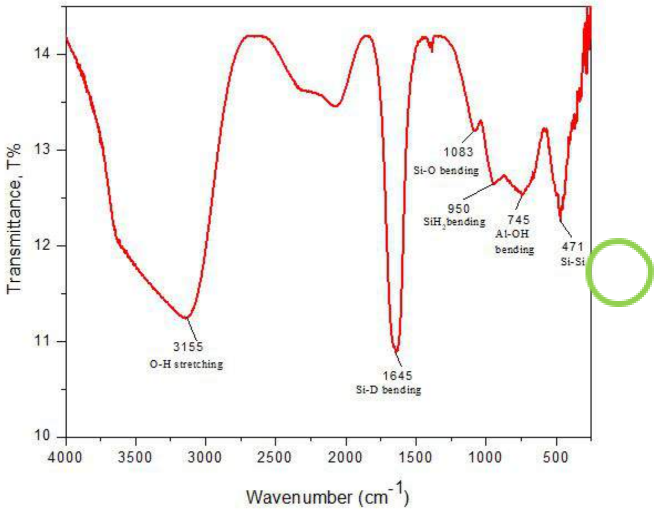

Fig.4: FTIR spectra of sample produced by Aluminothermic Reduction process.

Table II: Quantitative Result of Sample

Analyte	Result%	Proc-Calc	Line	net Int.	BG Int.
Si	54.1748	Quant. -FP	SiKa	46.246	1.252
Mg	25.9371	Quant. -FP	MgKa	12.098	0.075
Al	14.1006	Quant. -FP	AlKa	10.105	0.045

Ca	3.0006	Quant. -FP	K ka	4.237	0.114
K	1.6475	Quant. -FP	CaKa	2.270	0.095
Fe	0.4759	Quant. -FP	FeKa	2.491	0.082
Na	0.4511	Quant. -FP	NaKa	0.108	0.008
Ti	0.1145	Quant. -FP	TiKa	0.066	0.016
P	0.0751	Quant. -FP	P Ka	0.101	0.066
Mn	0.0229	Quant. -FP	MnKa	0.053	0.062

3.5 UV Spectroscopy Analysis

From the absorbance data, the absorption coefficient α can be calculated using Lambert law:

$$\alpha = 2.303 \times A/d \qquad (2)$$

Where, A= absorption, d= thickness.

The absorption co-efficient, α with photon energy. Fig.5 is shown the Absorption co-efficient (α) as function of photon energy (hν) of sample. The absorption coefficient α exhibits low values which means that there is a large probability of the allowed indirect transition. From Fig.6. the indirect band gap of sample produced by Aluminothermite process is 1.249 eV.

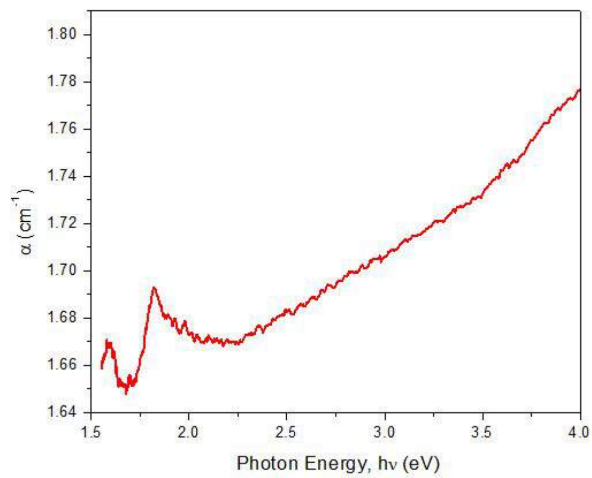

Fig.5: Absorption co-efficient (α) as function of photon energy (hν) of sample

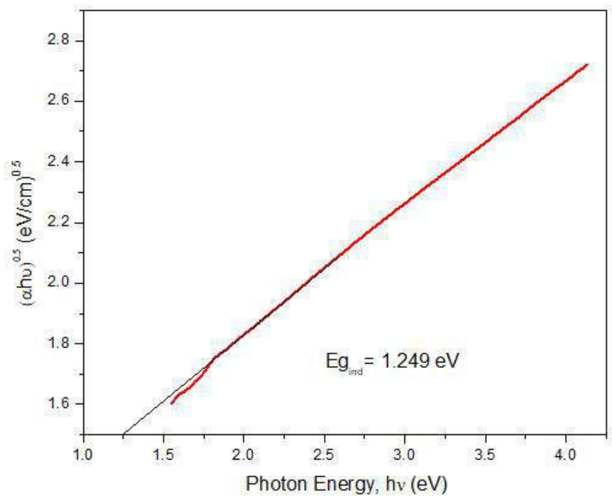

Fig.6: $(\alpha h\nu)^{1/2}$ versus photon energy (hν) of sample

IV. CONCLUSION

The present study was illustrated the extraction of silicon from sand which collected from the Padma River near Rajshahi city by Metal-Thermite reaction. Aluminum was used as metal of reduction reaction. From our study it was remarked various type of characterizations like XRD, SEM, FTIR, and XRF spectroscopy analysis. Observing XRD data, it was observed that the presenting element and the structure of prepared sample. In this process, Si (220), Si (200) planes were present and had FCC crystal structure. The particle size was 31.52 nm an average. SEM provided the surface appearance of sample, it was observed that the particles size was around 25 nm to 40 nm. It had been comparable to the getting result of XRD data. According to FTIR, it was seen that silicon-silicon bonding was presented. From XRF, it was found the percentage of silicon of given sample. It was observed that the prepared sample contained 54.1748% silicon. Others element likely Mg (25.94%), Al (14%) and other minor element was present. From UV spectroscopy analysis, the band gap of prepared silicon is 1.249 eV which is slightly deviated from standard due to presence of impurity. It is feasible to get better silicon in the further leaching process. The research is ongoing and trying to get better amount of silicon. We assume this silicon will suitable for fabrication of electronics devices.

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