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Photoluminescence Studies with Fresh Water Centric Diatom *Cyclotella Species*

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Abstract—*Over the last few years tremendous effort has been made in the development of silicon technology for applications in multitude of important areas ranging from bio-imaging to photovoltaic device fabrication. Recently porous silicon structures have gathered much attention due to their unique optical and electronic properties which sometimes exceed the performance of equivalent direct bandgap materials. Here we present an analysis of the optical structure and properties of a representative diatom Cyclotella species. From X-ray powder diffraction (XRD) pattern, amorphous structure of frustules was confirmed. Energy dispersive X-ray spectroscopy (SEM- EDS) spot analysis confirmed that diatom frustules were consisted of mainly silicon in the form of silica with 90% atomic weight. Uv-Visible (UV) absorption peak were obtained at 258 nm. Frustules of diatoms cleaned with HCl and H_2O_2 were examined by optical microscopy and Scanning Electron Microscopy (SEM).The microscopy images showed the nano sized pores of the frustules with regular arrangement. These nano sized pores were the main cause of the blue photoluminescence (PL), which was the fundamental property for any optoelectronic and photonic devices. The strong blue PL centered at 440nm indicated the diatoms to be a potential candidate for luminescent devices.*

Keywords*: diatoms, centric, porous structure, photoluminescence, hydrogenated amorphous silica.*

1. INTRODUCTION

Nanostructured optical devices attain more interest in various fields. In the last decades a large amount of work has been going on in developing well-ordered nano sized structures. But nature has already realized and optimized such structures [1]. These structures with excellent morphology [2], unique optical properties [3], and mechanical properties [4] have no comparison with man-made devices. One such fascinating example is Diatoms. They are a group of unicellular, eukaryotic, photosynthetic algae, whose protoplasm is enclosed by a micro shell made of nanoporous hydrogenated amorphous silica, called the frustule, formed by two valves interconnected by a lateral girdle. The frustules of diatoms can be regarded as biogenic photonic crystal [5]. The different families of diatoms can be classified into two main orders: Centrales, characterized by a radial symmetry of the frustules and Pennates, which are bilaterally symmetric In the recent past, substantial amount of research has been done to understand the way in which diatom frustules interact with light. For example, diatom structures are used as naturally made high- precision templates for nanoparticles assembly, as "living" three- dimensional photonic crystals [6]. Diatom frustules show visible photoluminescence emission which may represent the starting point for sensing applications based on optochemical transduction [3]. Diatom possesses weak green photoluminescence [7] and can act as a template for bioclastic material possessing [8].

In this work we studied the photoluminescence property of centric diatom frustules.

2. EXPERIMENTAL

2.1 Culture of Diatoms

Diatoms were collected from Niribili, Tezpur University, Sonitpur, Assam in the month of January 2015. The collected fresh water diatoms were cultured by using WC medium proposed by Guillard and Lorenzen [9] in the BOD incubator. To make the culture media slightly acidic we lowered the pH from 7 to 6.2.

The cleaned diatom frustules were added in conical flasks and put them in the BOD incubator for culture at 23.5°C for 30 days maintaining with 14 hours day light and 10 hours night cycle.

2.2 Sample preparation

In order to characterize and analyze the diatom frustules by Optical microscopy, Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), UV-Vis spectroscopy, Energy Dispersive X-ray spectroscopy (EDS) and Photoluminescence (PL) spectroscopy we needed a proper cleaning procedure to remove external organic part that covering the frustules and mineral debris (mud, sand, slit, leaves of aquatic plants, harvest from a plankton). We used acid treatment method to remove organic parts with some modifications for better results.

2.3 Optical microscopy analysis

Optical microscope is used to study about classification and morphology of microbiological organisms. The morphology of frustules of diatoms was analyzed by Axiostar plus optical microscope with 100x resolution.

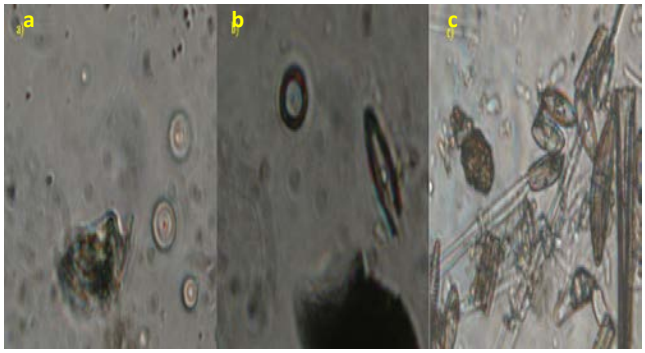


Fig. 1: (a), (b) & (c) show Optical microscopy images of diatoms in a biological colony with magnification 100x.

2.3 SEM/EDS analysis

A vast analysis of morphology of diatom frustules was done by JEOL JSM-6390LV scanning electron microscope and INCAx-sight energy dispersive X-ray spectroscopy (EDS) detector.

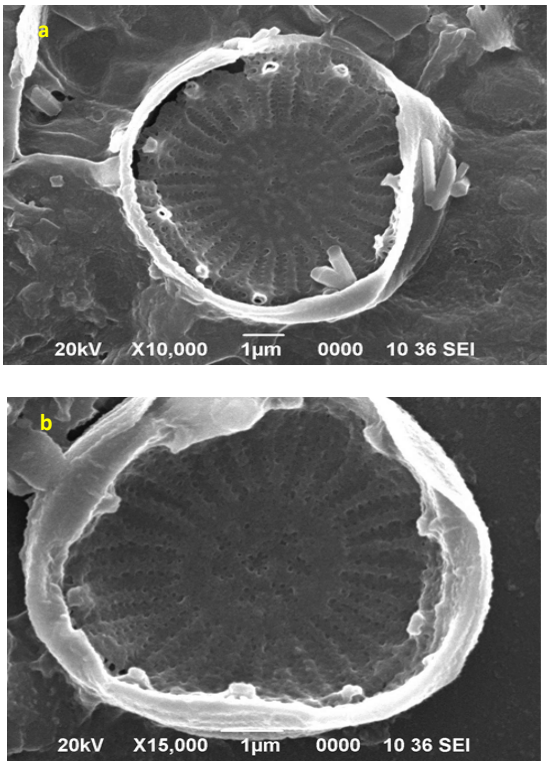


Fig. 2: (a) & (b) show SEM images of *Cyclotella species* of diatom frustules.

The geometrical patterns had been obtained by Scanning electron microscopy. Fig. 2(a) & (b) show the SEM images of whole valve of *Cyclotella species* of diatom frustules cleaned by hydrogen peroxide and hydrochloric acid. The total diameter of the cells was around 7.5 micrometer. The thickness of the silica slab was about 1micrometer. It has been reported that the cell-wall thickness decreases slightly from generation to generation [5]. SEM images showed frustules were composed of uniform distribution of nanopores. The formation of pattern is believed to proceed via self-organized phase separation [10]. In some portions, the pores were not clearly visible it was due to the presence of organic matter.

2.4 EDS spectrum of diatom frustules

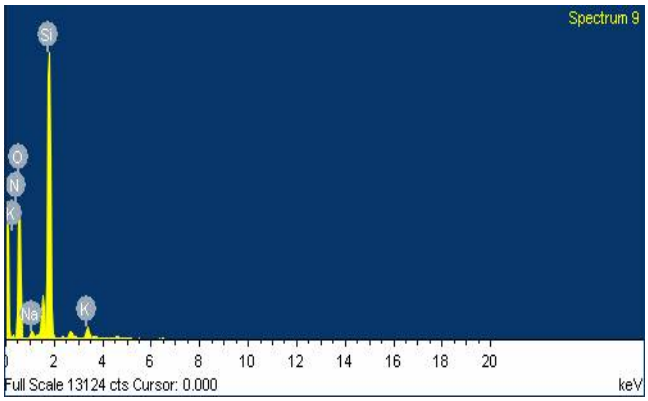


Fig. 3: Energy dispersive X-ray spectroscopy of diatom frustules.

Table 1

Element	Weight (%)	Atomic (%)
Si K	29.82	19.49
O K	59.76	68.55
Na K	1.12	0.90
N K	7.97	10.45
K K	1.33	0.62
Total	100.00	

The SEM-EDS spot analysis confirmed that diatom frustules were mainly composed of silicon in the form of silica (SiO2). The characteristic k-energy peaks were identified and tabulated in table 1.

3.5 XRD analysis

The XRD patterns were collected using a RIGAKU MINIFLEX diffractometer with Cu-Kα radiation (λ=1.5405 A^0).

The diatom frustules were subjected to XRD analysis to elucidate the chemical nature in the range of diffraction angle (2Θ) between 10^0 and 80^0. We did not find any distinct peaks, which confirmed that diatom frustules were amorphous in nature.

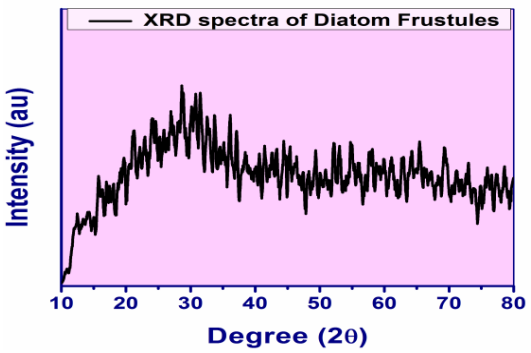


Fig. 4: XRD pattern of diatom frustules.

3.6 UV-Visible spectroscopy

Optical absorption spectra were obtained from a UV visible absorption spectrophotometer (UV 2450, Shimadzu Corporation). Absorption spectrometer works in a range from about 200nm (in the near UV) to about 800nm (in the very near infra-red).

As can be observed, the absorption peak of raw diatom frustules was in the UV region, maximum value located at 258 nm and then decreases with increasing the wavelength.

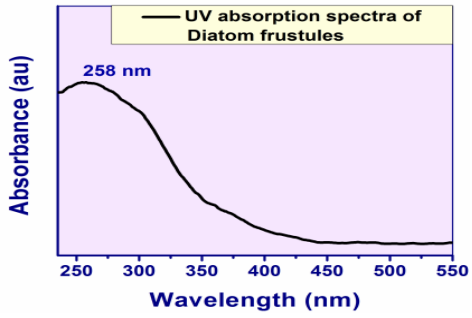
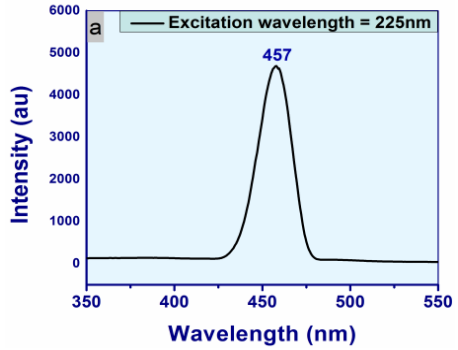


Fig. 5: UV absorption spectra of diatom frustules.

3.7 Photoluminescence (PL) spectroscopy analysis

The PL measurements were performed at room temperature using a Xe-lamp as the excitation source at 225nm and 250 nm and data were collected by a computer controlled standard monochromator based photodetection system (PERKIN ELMER LS 55 fluorescence spectroscopy).



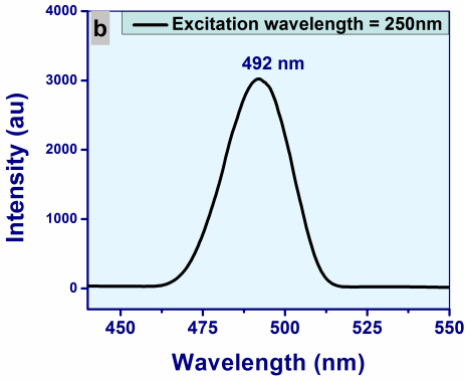


Fig. 6: (a) & (b) show PL emission spectra of diatom frustules centered at blue wavelength region when excited at 225nm & 250nm wavelength.

PL emission of diatom frustules was dependent on species and surrounding environment. According to previous studies the visible PL emission of diatom frustules was unclear [11]. It may be due to nano pores and metal oxides present in the frustules.

3. CONCLUSIONS

Photoluminescence emission properties of fresh water centric diatoms were studied. Isolated frustules were confirmed and identified through Optical microscopy and SEM analysis. Their chemical composition were studied by EDS spot analysis and resulted 90% of the species composed of silica and another 10% was composed of metal oxides. UV-Visible spectroscopy confirmed that frustules absorb energy in the UV region and emitted in the blue region resulted by PL emission when excited at 225nm and 250nm wavelength, which was not been observed in case of marine diatoms.

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