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Mineral analysis of an artificial pond sediment samples from the Western Cape Province, South Africa

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Mineralogical characterization was carried out to determine the major and minor constituent minerals present in sediment samples collected from five different locations at the Western Cape artificial pond sediment, South Africa by Fourier transform infrared (FT-IR) and X-ray diffractometry (XRD) techniques. The minerals were identified with the available literature from the IR absorption band of the locations of different peaks. The FT-IR technique was highly useful in identifying different minerals in sediments. The XRD analysis revealed that the minerals of artificial pond sediments in Western Cape are predominantly composed of quartz while carrolite was present in location one. The presence of pyrite could also suggest anoxia, which in turn will imply differential Fe (Iron) levels in the sediment as a function of the degree of anoxia in the sediment.

Key words: Pond sediment, peaks, characterization, FT-IR-XRD techniques, mineralogical.

INTRODUCTION

Sediment is an important reservoir of contamination and serves as the ultimate sink of elemental contaminants. Sediment quality is one of the factors that determine the environmental health of an aquatic system. The non-biodegradable features and easy accumulation of trace element and heavy metals make it more hazardous. Sediments are the principal carriers of the trace elements in the hydrosphere. Sediment particles are made up of materials derived from rock, soil, biological and anthropogenic inputs. The basic structural unit of inorganic sediment is silicate and aluminosilicate (Fatoki et al., 2012; Ravisankar et al., 2010). The main composition of sediments includes various silicate minerals (that is clay, quartz, and feldspar), iron oxide of

hydroxides (that is gibbsite) and carbonate (calcite, aragonite, and dolomite (Ravisankar et al., 2010). There are various ways of classifying sediments which are based on composition, particle size and source.

The characteristic features of sediments depend on particle size distribution, porosity, organic matter percentage and sorption capacity of pollutants. Mineralogy is a fundamental part of a lithological description but the analytical capabilities for accurate mineral analysis lag behind other parameters (Matheson and Herron, 1993). The mineral composition of lake, coastal and marine sediments is of broad interest because of its usefulness as an indicator of environmental change. According to the various studies on mineral distribution, it has been observed that the mineral distribution in the lake - environment morphology is indicative of the sediment source (Wang et al., 2006). The mineral analysis is the prominent area of research. It improves the economic growth of the country.

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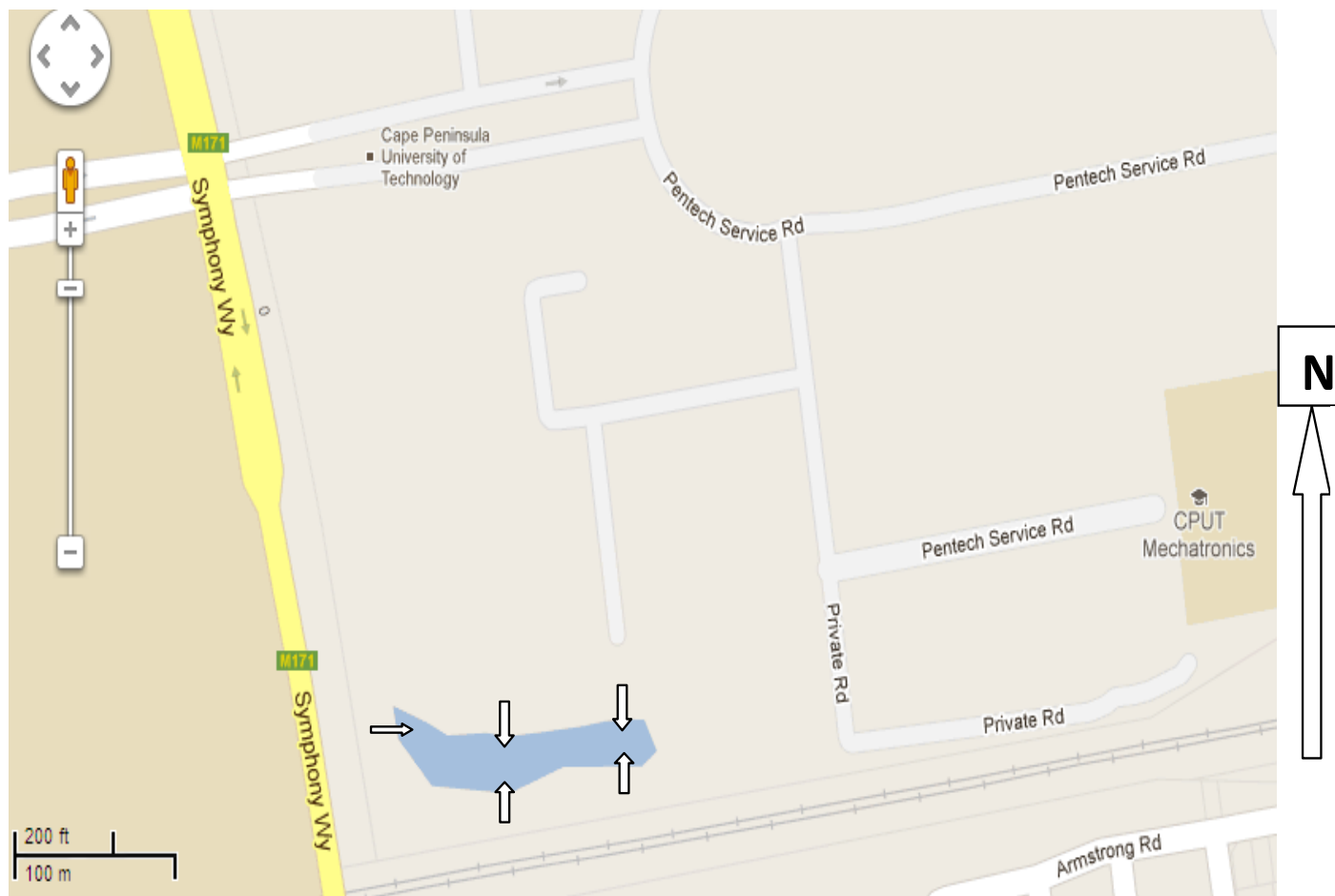


Figure 1. Map of the artificial pond on (S 33° 56 05", E 18° 38 22") showing the sampling point in Western Cape.

Radioactive minerals such as limonite, rutile, zircon, garnet etc are thus useful in nuclear reactors while quartz is used commercially as quartz crystal and talc powder.

There are different methods for mineral analysis which include differential thermal analysis, X-ray powder diffraction studies, ultraviolet-visible spectrometry, infrared spectrometry, etc. Fourier transform infrared (FT-IR) technique has the greatest advantages over other techniques for its rapid, cheap and non destructive capabilities. Exposure to pollutants has great effects directly or indirectly on the inhabitants. These effects are of great concern. The two sources of heavy metal contamination in lake, river and marine ecosystems are through natural and anthropogenic sources during the course of their transportation. In developing continent like Africa, lack of adequate control and proper planning has led to pollution of lake, river and sea water due to unlawful acts of dumping wastes as well as washing and bathing in open lakes. In the present study, a mineralogical investigation of sediments samples from artificial pond in Western Cape has been undertaken using FT-IR and XRD techniques. XRD has been used

to identify the mineral phases present in the sediment samples. The FT-IR and XRD were used for the identification of various chemical groups including functional group present in the mineral constituents of the sediments.

MATERIALS AND METHODS

Sample preparations

Samples were collected in triplicate from the surface layer (5 to 16 cm) along bottom of the lake. Grab samples were collected from five locations throughout the Western Cape Province, South Africa. The coordinate of the sampling points is: location 1 (S 33 56 05" E 18 38 22"), location 2 (S 33 56 05" E 18 38 22 E), location 3 (S 33 56 06" E 18 38 21") location 4 and 5 (S 33 56 06" E 18 38 18"). Figure 1 represent sampling points. The samples were placed in plastic poly Zip-block bags in an ice chest and transported to the laboratory. The laboratory samples were spread on aluminium foils and air dried at room temperature for a week. The dried sediments were grounded using a mortar and pestle, screened and sieved with a 500 µm, laboratory test homogenised and finally stored at 4°C in refrigerator prior to both FT-IR and XRD investigation respectively.

Table 1. IR absorption frequencies cm^{-1} of quartz, pyrites*, calcite**, kaolinite***, feldspar****.

PT 1	PT 2	PT 3	PT 4	PT 5
455	695	450	458*	455
591.98*	776.91*	600*		800
517.09*	587.16****	828**		517*
700.13**	823**			700.79**
734.53**				580****
829.37**				
1030***				

Reagents and instrumentation

MilliQ water 18.2 M Ω cm (Millipore Bedford, MA USA) from a MilliQ deionising system, was used for this study. All reagents used (that is acetone, potassium bromide powder (KBr) were of analytical grade. Perkin-Elmer Fourier transform (FT-IR) spectrum one Spectrophotometer instrument (made in USA) was used for the instrument analysis. The Spectrum one IR has a very large, purgeable sample compartment and it can operate in ratio, single beam, or interferogram mode. It is an optical system that gives data over wave number ranges 7800 to 370 cm^{-1} (220 cm^{-1} with CSI beam splitter) with Q best resolution of 0.5 cm^{-1} . It is equipped with an electronic system based on the Motorola DSP56303 Digital signal processor.

XRPD (X-ray powder diffraction spectrophotometer)

A Phillip PANalytical PW 3830/ 40 X-ray generator with a PW 3710 MPD control X-ray diffraction system using the Xpert data collector and identify software program were used for this investigation. The instrument operates at 40 kV and 25 mA, respectively and sufficient water pressure is maintained at 400 to 600 kPa. The instrument is coupled with curved Cu-filtered Cu-K radiation with slow scan speed of 0.040°/s with a continuous Goni scan mode.

Sample preparation for XRD and FT- IR analysis

For XRD analysis

This was used to determine the solid/mineral phases in the sediment. 1.0 g of the surface sediment (20 to 30 cm depth) was taken from each sample from the sampling point for analysis (no triplicate analysis involve here). The sediment samples for X-ray powder diffraction (PXRD) were ground to powder with a mortar and pestle at liquid nitrogen temperatures. The powdered samples were sieved to remove sand particles using a 40 μm mesh. The PXRD profiles were taken using an X-ray diffraction apparatus with Cu-K radiation (40 kV, 25 mA Rint-3900, sufficient water pressure was maintained at 400 to 600 kPa). The PXRD measurements were performed at room temperature with flowing cooled dry N_2 gas. The powdered sediment samples were introduced into a quartz glass capillary cell (2.0 mm diameter, 0.01 mm thick and 10 mm long). The PXRD profiles were acquired using a step width of 0.02° with a counting time of 0.040°/s step.

Infrared analysis

This was carried out using KBr method of preparation. Samples of

20 mg were mixed with 400 mg of spectroscopic KBr in the ratio 1:20 using a mortar and pestle. Before mixing, the required amount of KBr powder was dried at 120°C for six hours in an oven to prevent the broad spectral peak due to free OH from affecting the interpretation on the bond hydroxyls associated with any of the minerals. Also, the mortar and pestle were dried in an oven for 24 h before use. The mineral samples were weighed in a microbalance and placed in a clean gentle mortar along with the proper amount of dry KBr to prepare sample pellet. A pellet size that is 1 mm in thickness and 13 mm in diameter was prepared. The mixture was transferred to dye using a small camel's hair brush for pressing the pellet. Water and acetone were employed to clean the dye before preparation of another pellet. The prepared pellet was preserved in a moisture free glass container before it was placed in a suitable sample holder and introduced in the infrared beam for analysis. The IR spectra of the samples were prepared at room temperature. For quality control, the instrument was calibrated for its accuracy with the spectrum of a standard KBr powder. Prior to the spectrum of the sample being obtained; the spectrum of KBr powder was taken and checked for its accuracy.

RESULTS AND DISCUSSION

Table 1 summarises the absorption frequencies of the peaks in each of the spectra. The following minerals were confirmed when the observed frequencies were compared with the literature. They are: quartz, pyrite, calcite, feldspar and kaolinite. Quartz was reported in literature to be the second most dominant of all the minerals in the earth crust (Ravisankar et al., 2010). The chemical representation of quartz is SiO_2 . Quartz forms the major constituent of metamorphic, sedimentary, granite, limestone, etc. In the present study, it is confirmed that the most dominant of all the minerals present is quartz and from Table 1 its absorption band appears around 455 to 450, 798 to 800. This was similar to results recorded in literature (Coates, 1977; Ramasamy et al., 2003, 2004, 2005; Ravisankar et al., 2006).

Pyrites are principally present at locations 1, 2, 3 and 5. It can be observed from Table 1 that IR absorption frequency for pyrite ranges from 700 to 800. This could be connected to the presence of Fe at these locations; the source could be that the pond was receiving effluent from industry that treated its waste with ferric chloride.

Table 2. Major mineral phases in Western Cape artificial pond sediments (observed from XRD).

Location	Quartz	Pyrites	Calcite	Kaolinite	Feldspar
PT 1	•	•	•	•	
PT 2	•	•	•		•
PT 3	•	•	•		
PT 4	•				
PT 5	•	•	•		•

Calcite, this particular mineral, present principally across all locations, could be as a result of deposition of shell of dead planktonic life settle on the pond and also in evaporated setting. Feldspar was only present in location 2 and 5 while kaolinite was only present in location 1. All the mineral phase present are summarised in Table 2.

Conclusion

Qualitative mineral identification of Western Cape artificial pond was carried out using both the FTIR and XRD techniques respectively. The IR analysis indicated the presence of quartz, pyrite, calcite, feldspar and kaolinite. The XRD technique only confirmed the presence of quartz across all the locations and quartz was the most dominant of all the minerals. The diffractograms were similar for all the samples analysed. The presence of quartz in the entire sample was supported by XRD and this confirmed that both XRD and FTIR techniques were suitable in mineral analysis.

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