International Journal of Aquatic Research and Environmental Studies 4(1) 71-89 2024

Mineral and elemental compositions of suspended particulate matter (SPM) and sediments from major rivers in the Southeastern Nigeria

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Received: July 2023 Accepted: November 2023

Abstract

Mineral and elemental compositions of suspended particulate matter (SPM) and sediments from Great Kwa, Calabar, Cross River, Imo, and Qua Iboe Rivers in the Southeastern part of Nigeria were assessed in this study. SPM samples were obtained by the collection of water samples from the studied Rivers, passing the water through dried and weighed Whatman fiberglass filters and the sediments obtained were dried to a constant weight. Bottom sediments were obtained using Van Veen Grab sampler, transported to the laboratory, and air-dried at room temperature for seven days. These samples were subjected to Energy Dispersive X-Ray Analysis (EDX), X-ray diffraction analysis (XRD),and Scanning electron microscope (SEM). EDX analysis revealed that, high levels of Si and Al were common for all the studied samples. Silicate mineral was observed in all the sediments. XRD revealed that Quartz, clays, micas, hornblende, potassium-feldspars and mafics were the major minerals in the SPM and sediments. Quartz, gypsum and gibbsite minerals higher during the high than in low tide. Zircon, amphibole, pyroxene, biotite, pyrite, and iron hydroxide were idenfied by SEM in the samples.The mineral and elemental compositions of SPM and sediments from the studied rivers revealed and the pollution status established.

Keywords: Energy Dispersive X-Ray Analysis,X-ray diffraction analysis,Scanning electron microscope, Water pollution, Mineralogical study, SoutheasternNigeria

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Introduction

The quality of water available for utilization by human has direct effect on the health status (Lin *et al*., 2022; Luvhimbi *et al*., 2022). The quality of water in the Niger Delta area of Nigeria has been greatly affected by the anthropogenic factor especially the oil exploration and exploitation activities within the area (Ebong and Etuk, 2016; Ewim *et al*., 2023; Iwegbue *et al*., 2023). Reports have shown that prolonged human exposure to polluted water may results in water-related diseases such as dysentery, cholera, hepatitis, and diarrhea (Magana-Arachchi and Wanigatunge, 2020; Lin *et al*., 2022). High levels of toxic substances may have resulted in the extinction of some aquatic organisms within the oil producing communities of Nigeria (Bebeteidoh *et al*., 2020; Yin*et al*., 2022).The aquatic ecosystem has high tendency of being polluted since it receives contaminants from numerous sources including municipal discharges, industrial, agricultural, and domestic wastes (Akhtar *et al*., 2021). Elevated levels of metals in aquatic ecosystem are risky to all biological lives including human and is an indication of anthropogenic impact on the water quality rather than geological enrichment (Ebong *et al.*, 2006; Algül and Beyhan, 2020; Sojka and Jaskuła, 2022). Water sediments accumulate high levels of toxic substances mostly metals and subsequently release them into the water channel (Ebong and Etuk, 2016; Bartoszek *et al*., 2022).The natural geological process like weathering of rocks also have negative impact on the quality of water and sediments in aquatic environment (Baba and Gündüz, 2017; Yoshimura *et al*., 2021). SPM and sediments in the aquatic environment are affected by both the natural and human-induced factors and it has negative impacts on both human and aquatic lives (Baba and Gündüz, 2017; Shil and Singh, 2019). However, if the anthropogenic rate at which sediments are enriched in a water system is higher than the natural source, it could be an indication of pollution (Chen *et al*., 2014).

SPM is also a fundamental component of geochemical, geological, and biological sequence in the aquatic ecosystem due to its abundance, physicochemical properties, and mobility (Fernandes *et al*., 2019). SPM in aquatic environment consists of materials that do not sediment under some hydrochemical and hydrodynamic conditions and can be retained on a filter paper with porosity of 0.45 µm (Walch *et al*., 2022). SPM links the pelagic and benthic zones together in aquatic environment by sedimentation and re-suspension (Sutherland *et al*., 2022). In the aquatic ecosystem, SPM has the potential of absorbing dissolved trace metals because of its large surface area and reactivity (Li *et al*., 2019). Reports have shown that SPM carries majority of the solid contaminants from the soil environment to the aquatic environment (Zeng *et al*., 2019; Fan *et al*., 2021; 2023).

Studies have shown that the major components of SPM are mica, carbonate, phyllosilicates, plagioclase, quartz, potassium feldspar, and clay minerals as well as chlorite, kaolinite, montmorillonite, and illite(Sun*et al*., 2020; Kumari and Mohan, 2021). Clay minerals emanate mostly from the chemical and physical weathering of rocks and from the final product of soil genesis (Biscaye *et al*., 1997; Kumari and Mohan, 2021). Clay minerals are divided into four main groups namely: kaolinite, chlorite, illite, and smectite (Han *et al*., 2022). The kaolinite group is mainly found in the humid tropical climatic zones of South America, Africa, and Indonesia in ferrasols soils(Ouyang*et al*., 2021). The chlorites originate from mechanical weathering of rocks and are easily degraded during soil genesis. The illite group originates from the temperate and cold climate areas, mainly from Podzolic soils. Mica minerals originate mainly from direct mechanical weathering of igneous and metamorphic rocks. These clay minerals and quartz constitute the principal minerals of loess deposits (Pye and Blott, 2006; Chmielowska and Salata, 2020). Feldspar and mafic (mainly amphibole and pyroxene) minerals are derived from mechanical weathering of igneous and metamorphic rocks (Kloprogge and Wood, 2020; Macheyeki *et al*., 2020).

SPM also have some organic components such as microorganisms and organic particles including cellular debris, detritus, and extracellular polymers (Walch *et al*., 2022). Hence, it can affect the turbidity, colour, nutrient availability, and microbial load, clarity and availability of oxygen in aquatic ecosystem (Hope *et al*., 2020; Lednicka *et al*., 2022; Nair and Nayak, 2023). SPM controls eutrophication as well as the bioavailability and mobility of organic and inorganic substances in aquatic environment (Patrolecco *et al*., 2010; Vidmar *et al*., 2017; Gordon *et al*., 2020). Recent reports have indicated that SPM influences the presence of some emerging contaminants such as nanoplastics, nanoparticles, soots etc in aquatic ecosystem (Wagner *et al*., 2018; Li *et al*., 2019; Surette and Nason, 2019).

Sediments in water emanate from SPM and weathering of rocks, sediments are made up of and are consist mainly of calcite, clay minerals (kaolinite, montmorillonite, and illite), feldspar, and quartz in a silicate form (Saito*et al*., 2017; Tian *et al*., 2021). The other minerals found in river sediment included plagioclase, pyroxenes, muscovite, K-feldspars, biotite, and hornblende (Kimeli *et al*., 2021).However, the compositions of sediments depend solely on the mineral compositions of the mother rock. Silicates in sediments consist of mainly quartz, clays, micas, feldspars, and mafic minerals (Kimeli *et al*., 2021). Quartz is one of the most abundant minerals and essential constituent of many igneous, sedimentary, and metamorphic rocks (Yao *et al*., 2017; Del Lama and de Frascá, 2018). Quartz is a chemically stable mineral common in the soil environment but can be washed by runoff into the aquatic ecosystem (Baldermann *et al*., 2021).

Studies revealed that identifying the source and constituents of SPM and sediments is necessary for the establishment of the pollution status of aquatic system (Ma *et al*., 2015, Zuliani *et al*., 2022). Researches on the evaluation of minerals and metals in SPM and sediments have been carried out in countries outside Nigeria (Lučić *et al*., 2018; Zhang *et al*., 2018; Zeng *et al*., 2020; Gordeev *et al*., 2022). However, information on the geological and mineralogical of SPM and sediment in aquatic ecosystem in the study area are limited. Emphasis are more on the physicochemical, metals and microbial loads of water, SPM, and sediment samples from rivers within the region (Abu and Egenonu, 2008; Igbinosa *et al*., 2012; Ebong and Etuk, 2017; Ebong and John, 2021; Ehiemere *et al*., 2022; Izah *et al*., 2022; Iwegbue *et al*., 2023).

The elemental and mineral compositions of SPM and sediments from some rivers within the Southeastern part of Nigeria were examined in this study. The objectives of this study were (i) to assess the source of metals in the studied SPM and sediments, (ii) to identify the minerals present in the studied samples, (iii) to understand the relationship between minerals constituents of sediment and concentrations of metals in the studied rivers, (iv) to provide a comprehensive information on the pollution status of the rivers investigated, and (v) to reveal the contributions of natural rock weathering processes and anthropogenic activities on the minerals and metals of these samples.

Materials and methods

Study area

The areas covered during this research lie between longitudes 7°31’E - 8°23’E and latitude 4°33’N - 5°10’N in the South-Eastern section of the Niger-Delta Region of Nigeria (Figure 1). Three (3) estuaries (Imo, Qua-Iboe, and Cross Rivers) and two (2) tidal rivers (Calabar and Great Kwa Rivers) were investigated in this study. The coordinates of the studied locations as obtained from the Geographical Positioning System (GPS) are in Table 1. The area investigated is a sedimentary basin consisting of Quaternary and Pleistocene sediments. The studied rivers are within the meso-tidal setting where the influence of tide and waves are experienced(Boboye and Akinmosin, 2018). The study area is within the subequatorial climatic zone in the humid tropical rainforest area (Hassan *et al*., 2020). The studied area is characterized by two prominent seasons namely: Dry season which lasts from November to March and Wet season that starts from April to October (Adejuwon, 2012; Murtala *et al*., 2020). The atmospheric temperature of the area is at its highest in February and lowest between July and september (Kemela and Phoebe, 2021). The humidity peaks in July while the minimum level is observed in the month of January (Ebong *et al*., 2020).

Sample collection and treatment

Water and sediment samples were obtained from Great Kwa, Calabar, Cross River, Imo, and Qua Iboe Rivers at high and low tides. The collection of water and sediment samples was done between November 2011 and February 2012. Water samples were obtained from the rivers investigated from the upper 20cm of the water channel in a 25-liter plastic containers free oil (Moussa *et al*., 2022). Samples collected were homogenized and filtered through already dried and weighed Whatman fiberglass filters with a porosity of 0.45 mm. Sediments collected on the filter paper were dried again at 105°C for 90 minutes and re-weighed to achieve the desired SPM (Zhang *et al*., 2014). The SPM obtained were stored in a cooler for further analyses. Bottom sediments were collected at the various rivers investigated at a depth of 0-5 cm using a Van Veen Grab sampler, transferred into nylon bags and transported in a cooler to the Laboratory. These sediment samples were air-dried at room temperature for seven (7) days (Rzetala *et al*., 2019; Mackowiak and Perdrial, 2023).

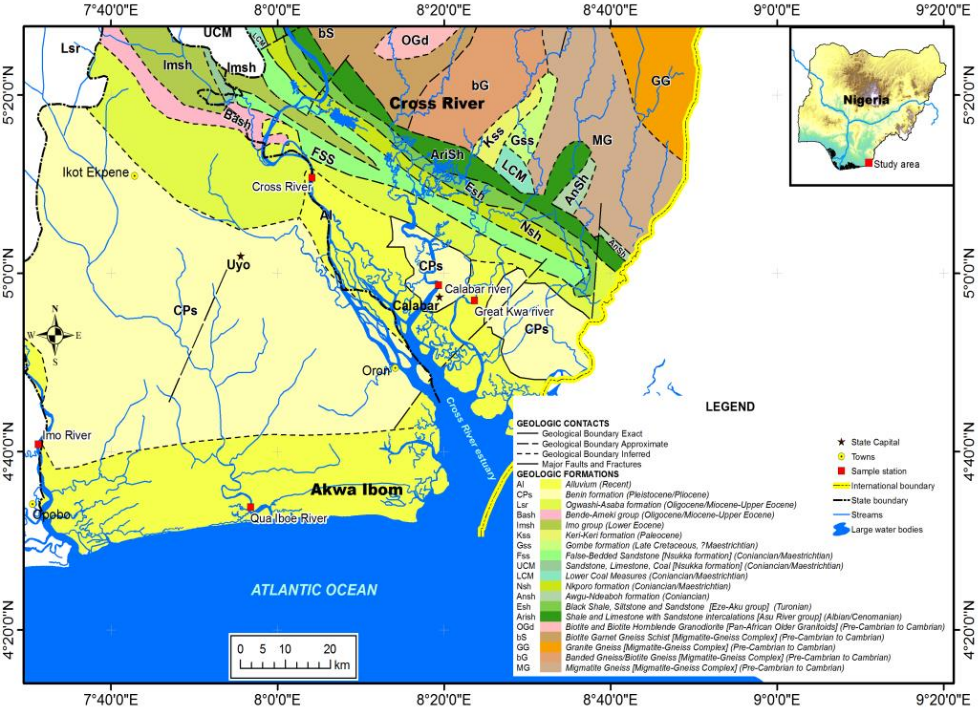

Figure 1: Geological map of the study area showing sample locations.

Table 1: Site location and Coordinates.

S/N	River System	Longitude	Latitude	Location
1	Great Kwa River	8° 23' 40.8516" E	4° 56' 57.9516" N	Apabuyo Bridge
2	Calabar River	8° 19' 21.9108" E	4° 58' 38.9208" N	Near Former CALCEMCO
3	Cross River	8° 4' 8.0112" E	5° 10' 43.1400" N	Calabar Itu Bridge
4	Imo River	7° 31' 15.8196" E	4° 40' 49.8216" N	At CrossRiver/Akwa Ibom Bridge
5	Qua Iboe River	7° 56' 46.2084" E	4° 33' 46.2384" N	Near Qua Iboe Town

Mineralogical analyses

The SPM and sediments obtained from the studied aquatic ecosystems were subjected to elemental and mineralogical compositions analyses as shown below. Quantitative compositional analysis of the samples was performed using a four spectrometer Cameca SX50 electron microprobe with a 10 nA beam current and accelerating voltage of 15 kV following the methods of Chebotarev *et al*. (2022) and Pérez-Huerta *et al*. (2023).

Quantitative analyses were performed with wavelength-dispersive spectrometers (WDS) after standardization with properly characterized compounds. Qualitative analyses (spectra) were obtained using an Imix Princeton Gamma Tech (PGT) energy dispersive system (EDS) using a thin-window detector (Stevenson *et al*., 2000; Mackowiak and Perdrial, 2023). Mineral compositions of the samples were obtained using Energy Dispersion X-Ray Analysis (EDX) according to the methods of Han *et al*. (2022). Scanning electron microscope (SEM) uses a beam of high-energy electrons to generate signals at the surface of the solid specimen. Signals derived from the electron-sample interactions shows the orientation of the materials making up the sample (Han *et al*., 2018; Lučić *et al*., 2019).

All mineralogical measurements were done with X-ray diffraction (XRD) analysis on a Siemens D8 X-ray diffractometer with a Cu Kα radiation over a 2θ range of 5 to 60°, a step interval of 0.02°, and duration of 4s step^{-1} (Kravchishina and Dara, 2014; Ali *et al*., 2022).

Results and discussion

Elemental and mineral compositions of suspended particulate matter and sediment from the studied rivers

Results of the compositions and levels of elements in sediments from the five (5) rivers investigated are indicated in Figures 2–6 below. Figures 7 and 8 illustrate the *mineralogical studies in SPM and sediments, respectively. Mineral compositions of sediment from* Great Kwa, Calabar, Cross River, Imo, and Qua Iboe Rivers are illustrated in Figures 9–13.

The elemental compositions of sediments from the studied rivers revealed the following: Samples from Great Kwa River showed high levels of Si, Al, Ca Mg and Fe with traces of Ti, Na and K (Fig. 2). Samples from Calabar River composed of high concentrations of Si, Al, K, and Fe with low levels of Mg (Fig. 3). Figure 4 indicates that sediments from Cross River contained elevated levels of Si, Al, Ca, K, Mg, and low concentration of Ti. High levels of Si, Al, and Fe with low levels of K and Ti were recorded for sediments from Imo River (Fig. 5). Qua Iboe River samples had elevated levels of Si, Al, Mg, and Fe but low concentrations of Ti and Ca (Fig. 6). The high concentrations of Si and Al reported in sediments from all the rivers investigated is consistent with the results obtained by Krivtsov *et al*. (2020).

EDX analysis revealed that majority of the particles identified in the studied sediments were mainly silicate minerals (quartz, clays, micas, feldspars, and mafic minerals (Mg-Fe) silicates) (Kimeli *et al*., 2021).

Results of the X-ray diffraction analysis (XRD) for the mineral compositions in suspended particulate matters (SPM) and sediments are shown in Figures 7 and 8, respectively.

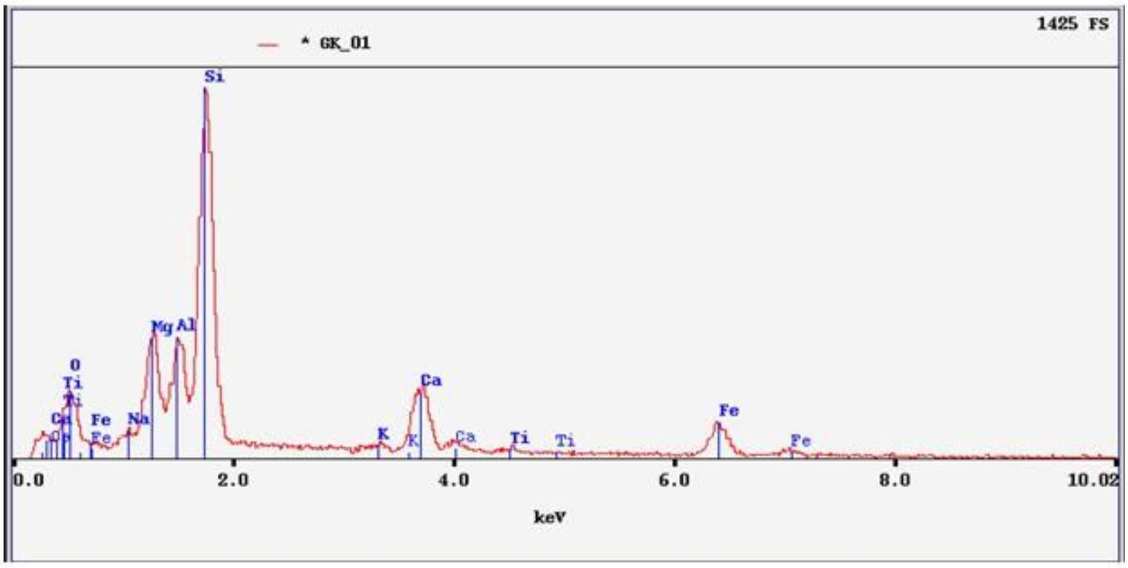

Figure 2: EDX spectrum of Great Kwa River sediment.

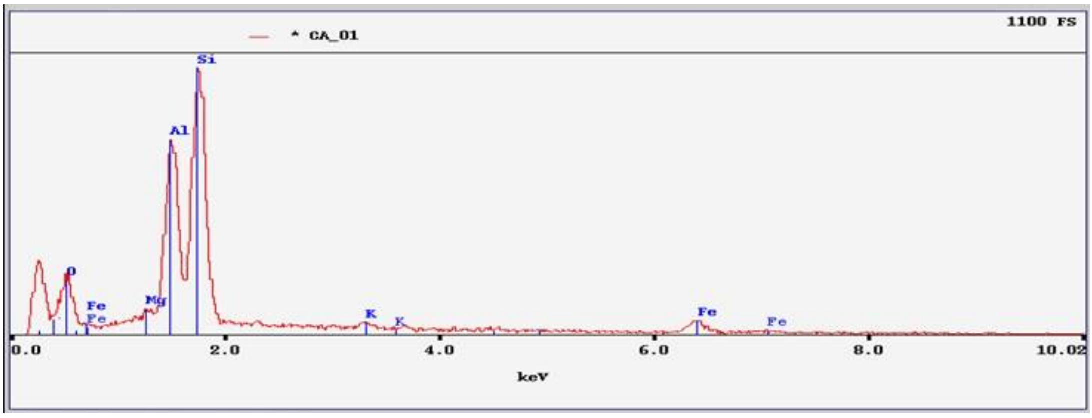

Figure 3: EDX spectrum of Calabar River sediment.

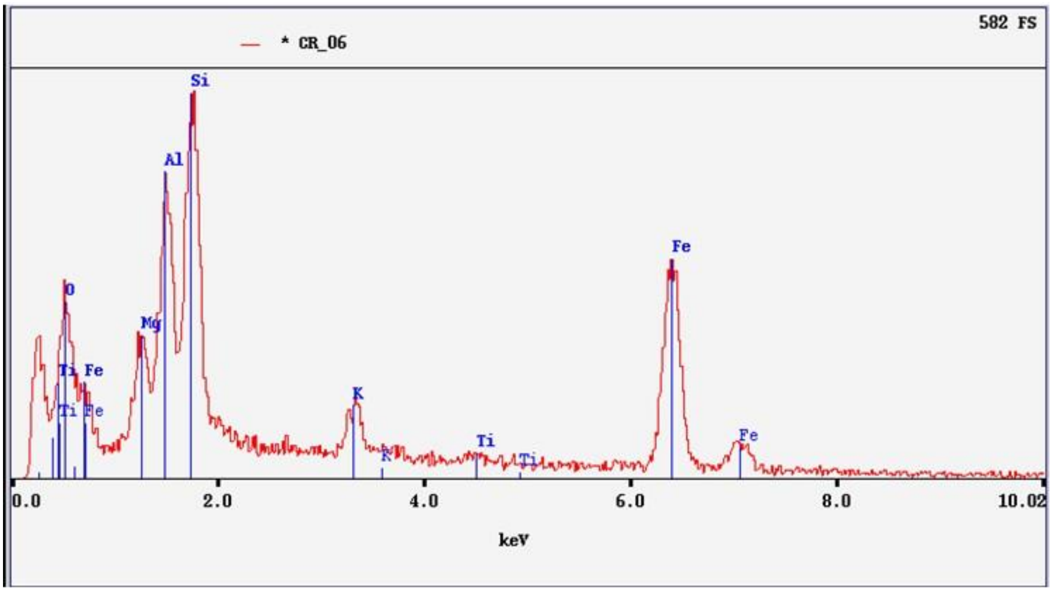

Figure 4: EDX spectrum of Cross River sediment.

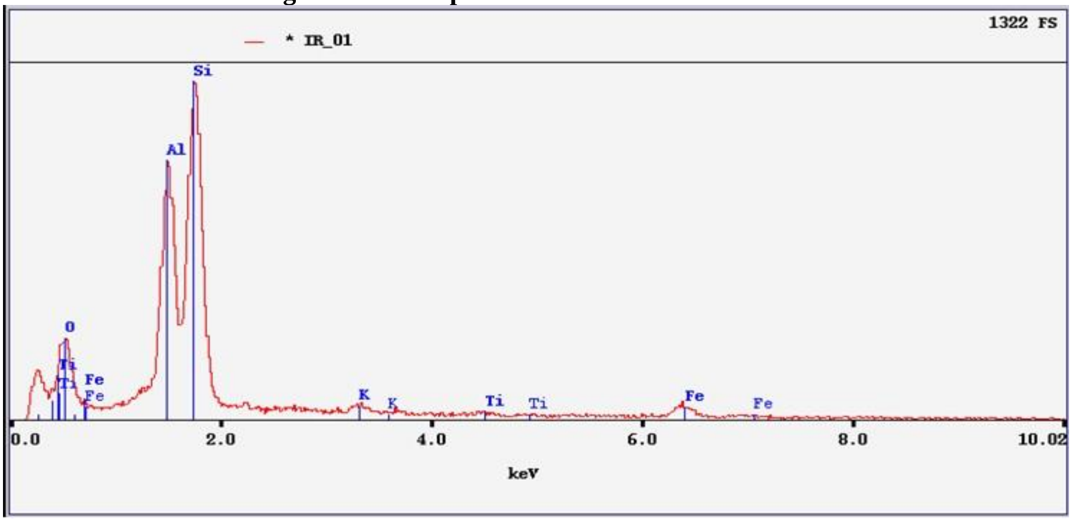

Figure 5: EDX spectrum of Imo River sediment.

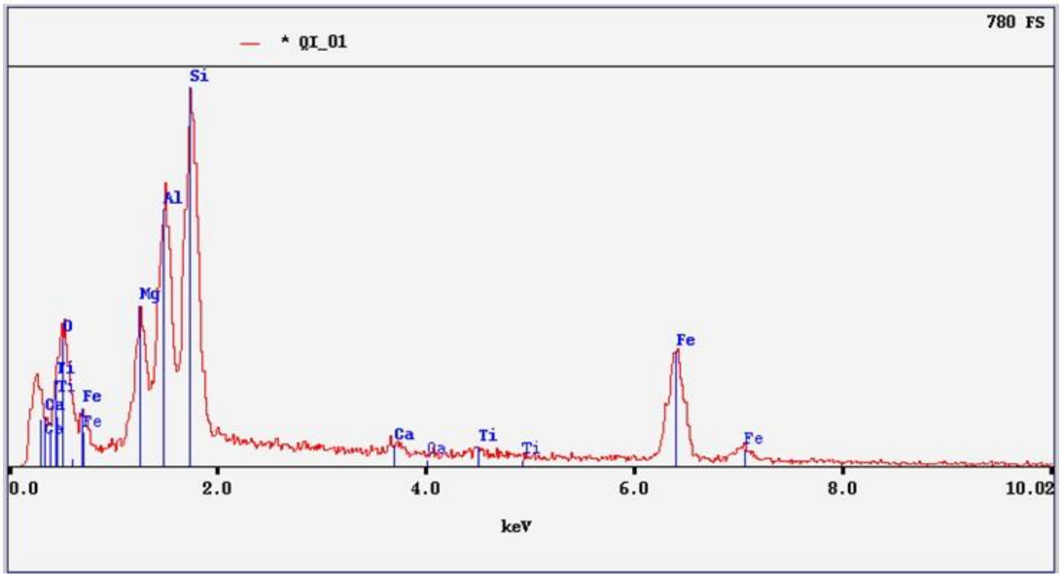

Figure 6: EDX spectrum of Qua Iboe River sediment.

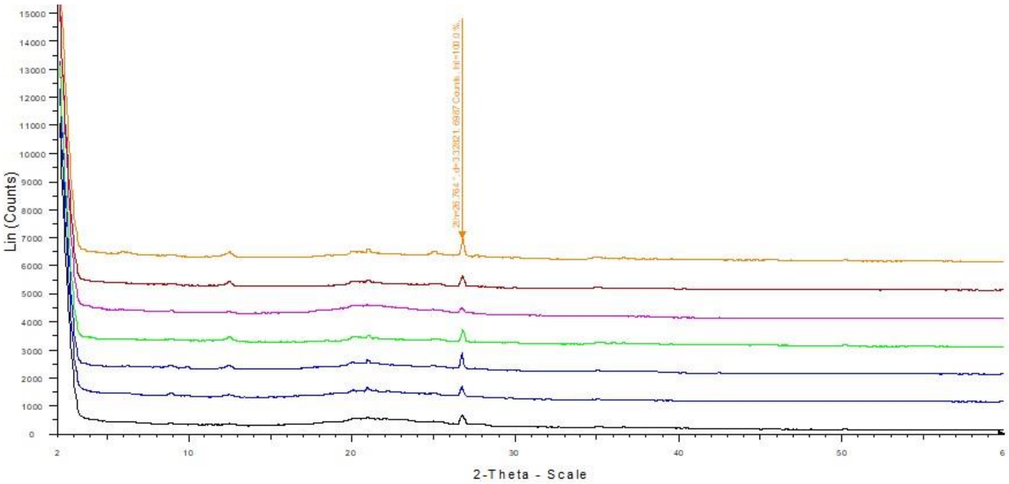

Figure 7: The XRD pattern of the suspended particulate matter. Blue = Great Kwa River; Purple = Calabar River; Green = Cross River; Dark red = Imo; River; Black = Qua Iboe River.

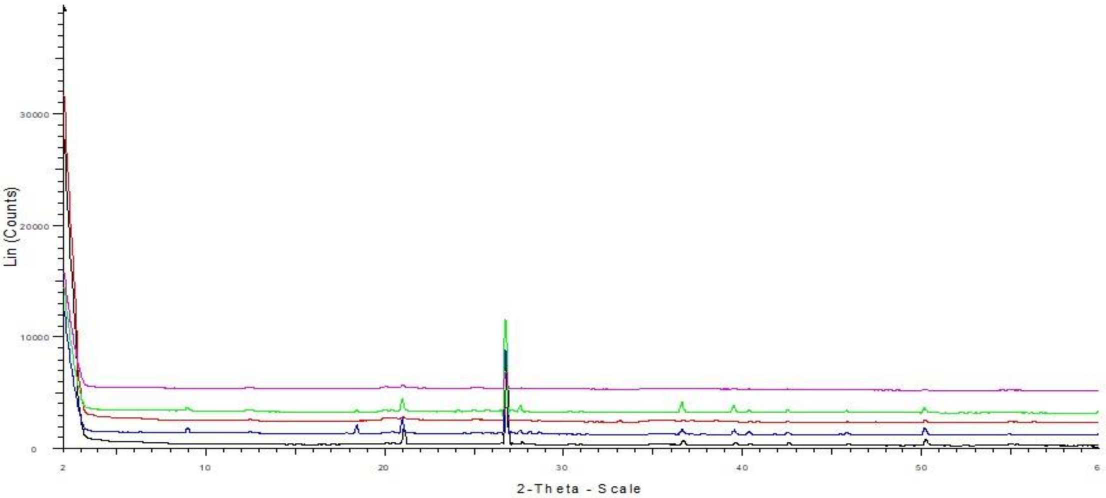

Figure 8: The XRD pattern of the sediment samples. Blue=Great Kwa River sediment; Purple=Calabar River sediment; Green=Cross River sediment; Red=Imo River sediment; Black = Qua Iboe River sediment.

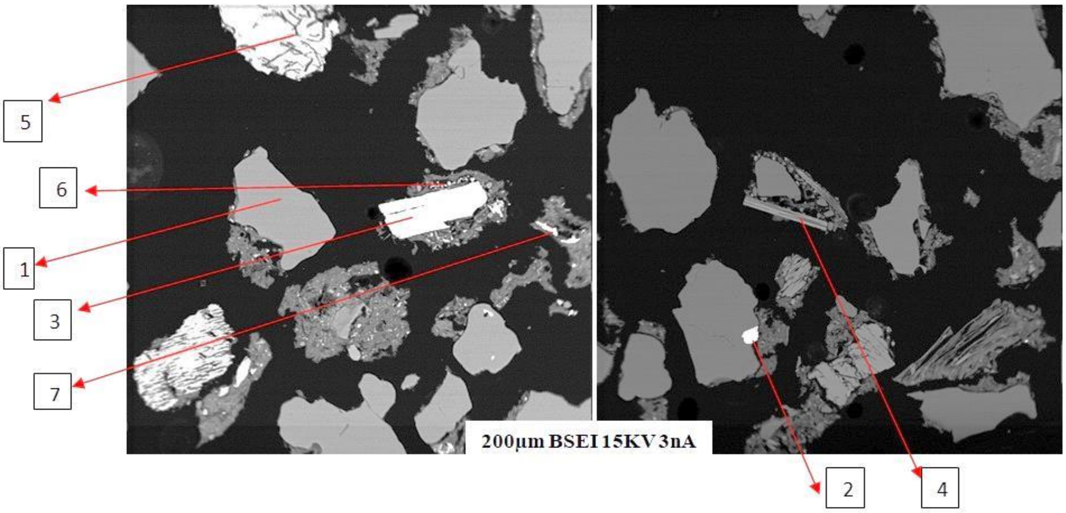

Figure 9: SEM image of Great Kwa River sediment. 1- Quartz; 2- Zircon ($ZrSiO_4$); 3- Amphibole/pyroxene; 4- Biotite (mica minerals); 5- k-feldspar; 6- Pyrite (FeS_2); 7- FeOH.

These Figures reveal that both SPM and the sediment samples were dominated by quartz (28.56%). This is similar to the results obtained by Lučić *et al*. (2019) and Jian*et al*. (2020) in their studies. Quartz was identified by the diffraction peaks at 4.21, 4.23, 3.33, 3.23, 2.44, and 1.81Å. As shown in Figure 7, the reflection peak at 3.33Å is more intense in samples from Great Kwa River than in other sediments. Hornblende with relative abundance of 20.11% was identified by the reflection peaks at 9.94, 9.91, and 8.34Å. Hornblende was detected mainly in the samples from the Great Kwa, Cross River, and Calabar Rivers.

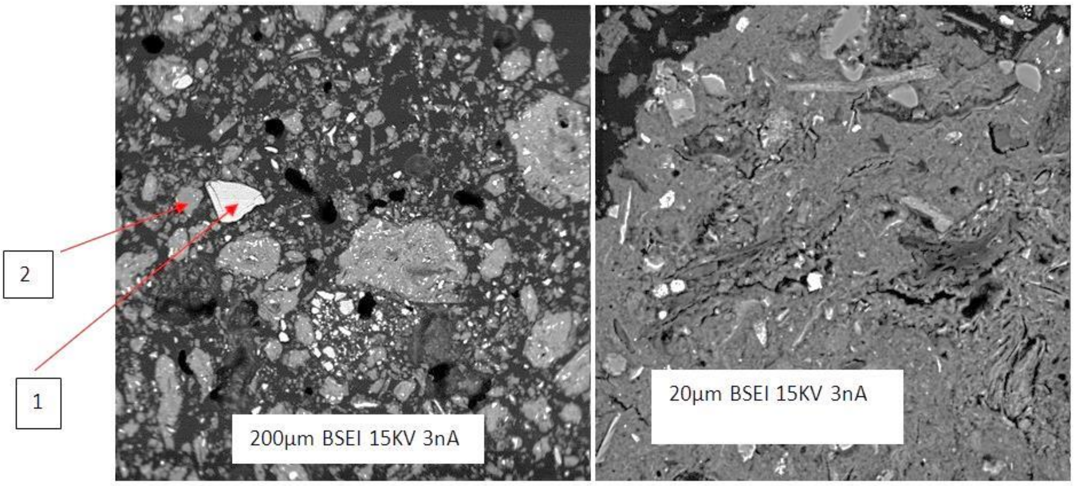

Figure 10: SEM image of Calabar River Sediment. 1- Quartz; 2 - Clay mineral (kaolinite, illite, FeO).

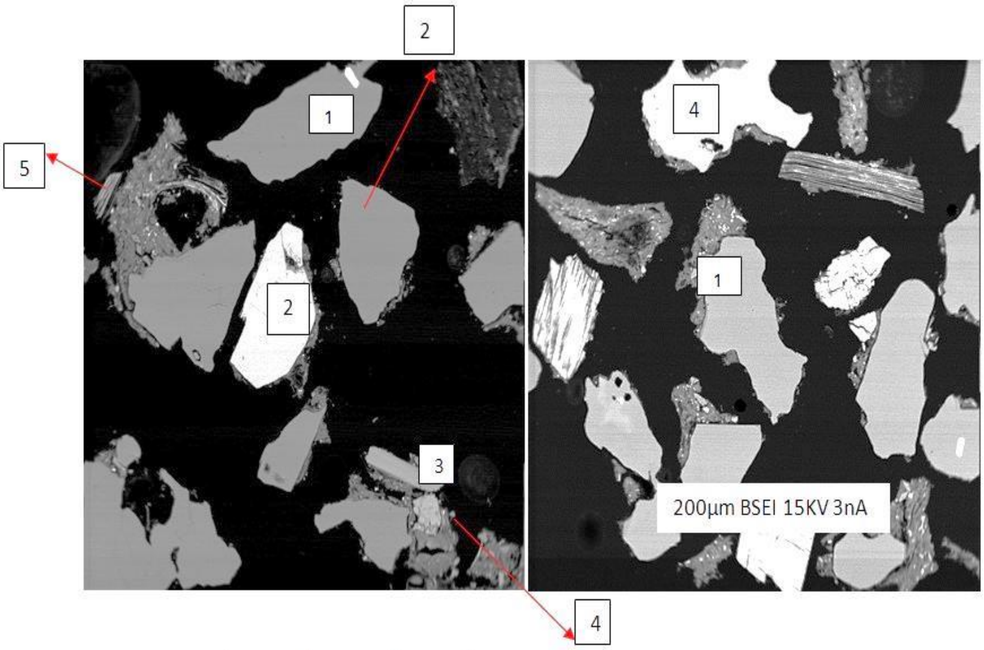

Figure 11: SEM image of Cross River Sediment. 1- Quartz; 2-Pyroxene; 3-White mica (muscovite); 4- K-Feldspar; 5-Mafic minerals.

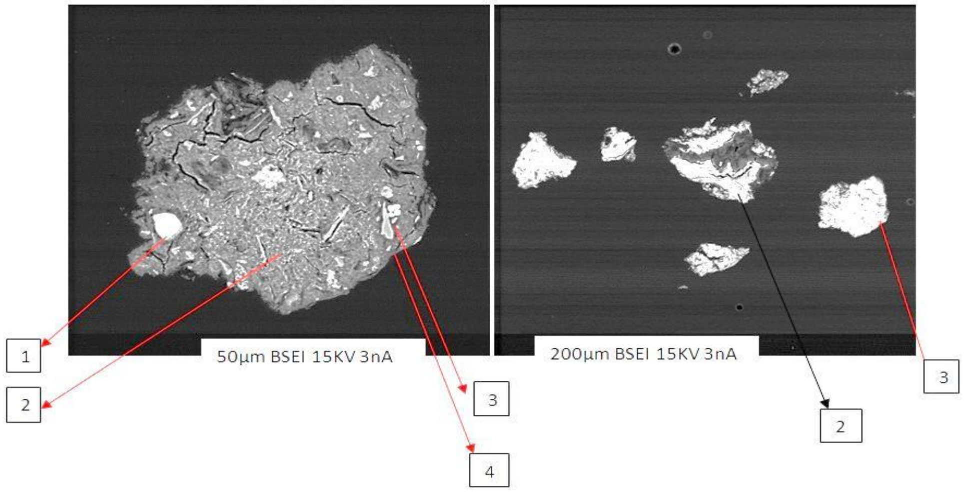

Figure 12: SEM image of Imo River. 1- K-feldspar; 2- Clay mineral (kaolinite, illite, FeO); 3- FeS_2 (pyrite); 4-Na-feldspar.

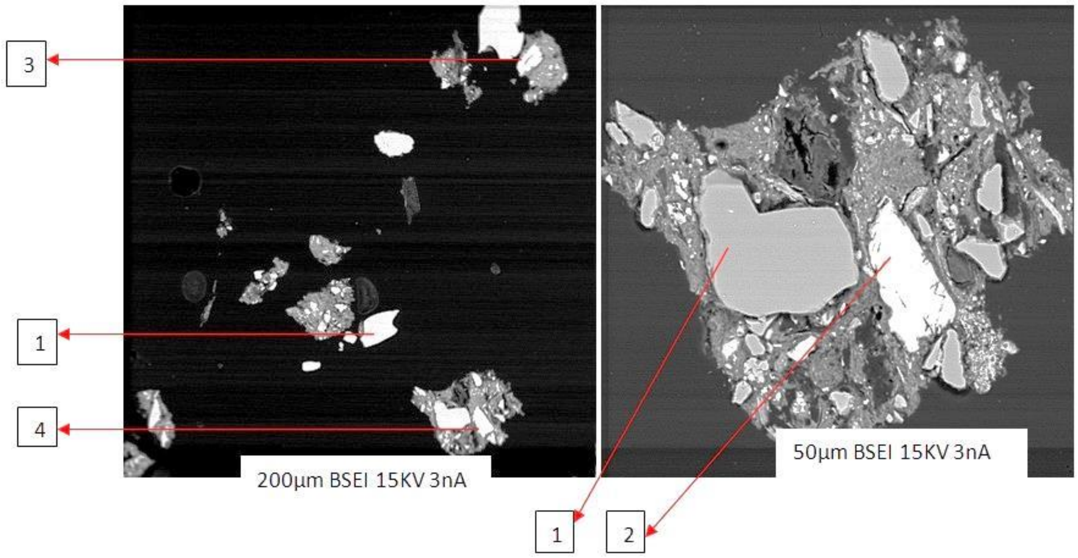

Figure 13: SEM image of Qua lboe River. 1- Quartz; 2- K-feldspar; 3- Clay mineral (kaolinite, illite, FeO); 4- Chlorite.

Gypsum (15.31%) was identified by the reflection peaks at 7.11, 2.88, 2.27, and 2.23Å. Gibbsite was identified primarily by reflection peaks at 4.79 and 4.81Å. Gibbsite constituted about 15.55% of the entire minerals found in samples from Qua Iboe and Great Kwa Rivers, similar to the results obtained by Sidibe and Yalcin (2019). Clay minerals (illite (2.7%), kaolinite (3.8%), and chlorite (3.8%) were identified by their characteristics basal X-ray diffraction peaks at 2.54, 4.44 and 4.97Å, respectively. Calabar and Cross Rivers samples contained traces of calcite (3.3%) and was identified by the reflection peaks at 2.94Å. Potassium feldspar with relative abundance of 12.10% and was identified by reflection peaks at 3.69, 3.55, and 3.46Å.

The XRD results revealed that apart from Quartz, clays, micas, feldspars, and mafic minerals identified by EDX, other minerals such as gypsum, gibbsite, and hornblende were also components in the studied SPM and sediments. This is in agreement with the results obtained in a study within Sava River in Croatia by Lučić *et al*. (2018). This result is consistent with the morphological characterization done on the samples with scanning electron microscope and energy dispersive X-ray analyzer. The XRD pattern obtained indicated that sediment samples were more crystalline than suspended particulate matter. The pattern also revealed that, samples obtained during high tide contained more quartz, gypsum and gibbsite minerals than the low tide samples. This is consistent with the report obtained in dust micro particles by Gryttinget *et al*. (2022). Quartz was the most common type of silica minerals in all the samples examined as indicated by the intense Si peak in EDX spectra. The grain surface characteristics (Conchoidal fractures and V-shaped pits) observed in the SEM micrographs (Figs. 2-6) distinguished quartz from other minerals identified (Madhavaraju *et al*., 2021). Quartz is one of the most abundant minerals and it is an essential constituent of many igneous, sedimentary, and metamorphic rocks (Yao *et al*., 2017; Del Lama and de Frascá, 2018). Quartz mineral emanates from the mechanical and chemical weathering of quartz rich rocks, and as a chemically stable mineral, it is common in the earth soil environment can easily be washed into the aquatic environment (Baldermann *et al*., 2021). This may have resulted in the observed abundance in the suspended particulate matters and sediments from the studied Rivers.

The scanning electron microscope (SEM) images of sediments from Great Kwa River in Figure 9 indicate quartz, zircon, amphibole, pyroxene, biotite (mica mineral), potassium feldspar, pyrite, and iron hydroxide as the major minerals. Calabar River sediments contained mainly quartz and clay minerals with many organic matters (Fig. 10). Quartz, pyroxene, white mica, potassium feldspar, and mafic minerals were the dominant minerals in sediments from Cross River (Fig. 11). Potassium feldspar, clay minerals, pyrite, and sodium feldspar were the principal minerals sediments from Imo River (Fig. 12). SEM images of sediments from Qua Iboe River in Figure 13 indicate quartz, potassium feldspar, clay minerals (kaolinite, illite, ferrous oxide (FeO) and chlorite as the major minerals. This is consistent with the report by Kumari and Mohan (2021) in a similar study.

Conclusion

The study revealed the mineral and elemental compositions of SPM and sediments from Great Kwa, Calabar, Cross River, Imo, and Qua Iboe Rivers, Nigeria. EDX analysis indicated the elements present in sediment from each river however; Al and Si were the common ones. It was observed that, most of the particles in the studied sediments were silicate minerals. XRD analysis on SPM and sediments from rivers investigated were dominated by quartz. The XRD results revealed that gypsum, gibbsite, and hornblende were also major components of the studied SPM and sediments. Scanning electron microscope (SEM) images of sediments from the different rivers studied showed varied minerals for the different locations. However, the common minerals in sediments from the various rivers studied were quartz and clay minerals. The study has shown that apart from anthropogenic factor, the natural geological processes in the aquatic environment can also contribute significant amounts of contaminants/pollutants to the studied rivers. The results obtained revealed the mineral and elemental compositions of the studied SPM and sediments.

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