

ORIGINAL ARTICLE

Mineralogical Characterization of Riverbed sediments from North-Eastern Tanzania: An Implication to their provenance

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ABSTRACT

We determined the mineralogical composition of riverbed sediments from six rivers located in North-eastern Tanzania. Both bulk sediments and separated clay fractions were characterized by x-ray diffraction (XRD) and micro-Raman spectroscopy (MRS) to identify the mineral components of the riverbed sediments. The MRS measurements were done by using the visible and near infra-red lasers with varied power and regulated exposure times for individual particles. The minerals were identified on the basis of their characteristic Raman spectra obtained from the 514.5 nm and 785 nm laser excitations. The X-ray diffraction analysis identified a few clay species, including illite, kaolinite and smectite in nearly all clay samples. A number of minerals were also identified from the bulk samples, but the most common were quartz, albite, muscovite, orthoclase and zeolite. The MRS spectra bands showed that sediments from all locations were characterized by quartz (SiO_2) and other different kinds of minerals. The minerals frequently identified were calcite (CaCO_3), aragonite (CaCO_3), spessartine ($\text{Mn}_3\text{Al}_2(\text{SiO}_4)_3$) and pyrolusite (MnO_2). Others were anatase (TiO_2), orthoclase (KAlSi_3O_8), rhodocrosite (MnCO_3), richterite ($\text{Na}_2\text{CaMg}_3\text{Fe}_2(\text{Si}_8\text{O}_{22}(\text{OH})_2$), rutile (TiO_2), illmenite ($\text{Fe}^{2+}\text{TiO}_2$), sanidine ($(\text{K}, \text{Na})(\text{Al}, \text{Si})_4\text{O}_8$) and to a lesser extent dolomite ($\text{CaMg}(\text{CO}_3)_2$), biotite, ($\text{K}(\text{Mg}, \text{Fe}^{2+})_3[\text{AlSi}_3\text{O}_{10}(\text{OH}, \text{F})_2]$), augite ($(\text{Ca}, \text{Na})(\text{Mg}, \text{Fe}, \text{Al}, \text{Ti})(\text{Si}, \text{Al})_2\text{O}_6$), kaolinite ($\text{Mg}(\text{Si})_2\text{O}(\text{Si})_2(\text{OH})_4$), sphene (CaTiSiO_5) and albite ($\text{NaAlSi}_3\text{O}_8$). The MRS technique was found to be particularly useful as a fast, non-destructive tool for determination of local components which cannot be characterized by other means. This investigation shows that riverbed sediments from the location are composed of a wide range of mineral phases from the rich geologic diversity of the area. The study gives initial insights and forms a basis for more comprehensive study in the future.

Keywords: Rive-bed sediments; mineralogical composition; micro-Raman Spectroscopy; Pangani River Basin; Tanzania

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INTRODUCTION

Sediment profiles are known to be closely related to the geochemistry of the river catchment in the area, including the rocks and soils from which they are eroded (1, 2). Studies have long established that levels of observed sediment-bound chemical species in an environment may be a function of both anthropogenic inputs and geochemical patterns from different compounds and minerals present naturally in the sediments (3, 4), although sediment qualities of many of the world's rivers are said to have been altered markedly by human activities.

It is also established that metallic oxy-hydroxides such as goethite and gibbsite which accumulate in soils from erosion and weathering of rocks get transported from the soils into nearby rivers and accumulate in sediments (5). This indicates that at some point the concentrations of metal contaminants from human activities may be masked by those occurring naturally in rocks and soils. Recent studies have stressed the fact that sediments mineralogical compositions and their relationship to the chemical composition may provide valuable insights into the origin and dynamics of different chemical species and river-derived materials found in an area (6). As such, techniques that can characterize molecules of compounds and provide information about the mineral phase of the given elements and identify natural compounds residing in the sediments are well established (7, 8, 9).

The Pangani River traverses a diverse geological environment from Mount Meru and Kilimanjaro down to the coast of Indian Ocean. The geology traversed varies from Neogene alkaline volcanic, in areas close to Mount Meru and Kilimanjaro, crystalline metamorphic rocks of the Mozambiquan belt at the middle of the

basin to fluvial and deltaic sediments in areas near the coast of Indian Ocean. Most rocks in basin area are obscured by superficial deposits composed of alluvial sediments and soils, products of weathering of underlying rocks (10, 11).

Most studies on sediments documented in the Tanzanian environment involve the detection, identification and quantification of contaminants of concern as an important tool for monitoring anthropogenic inputs in aquatic environments. However, this alone does not provide a full picture of what is going on at the site. Moreover, there are generally few documented works on mineralogical composition of fluvial sediments in African rivers (12, 13). Such lack of information is especially pronounced in Tanzania, where studies on sediment characterization are generally few.

The main objective of this study was to characterize riverbed sediment from six rivers located in Northeastern Tanzania so as to identify their mineralogical composition and relate them to the rock sources and fluvial inputs. In this work, x-ray diffraction was chosen as an analytical method that allows fast determination of mineral composition in a sample and micro-Raman spectrometry for the identification of materials on the basis of their molecular vibrational spectra, obtained by excitation with a laser light (14, 15). This study is the first application of the micro-Raman spectroscopy technique in the characterization of sediments in this the area, and probably one of the very few in the Tanzanian environment in general.

METHODOLOGY

Description of the study area

Tanzania is located in East Africa between latitudes 1°– 12° S and longitudes 29°– 41° E, bordered by Kenya and Uganda to the north and Mozambique to the south. The north-eastern part of Tanzania includes four administrative regions of Manyara, Arusha, Kilimanjaro and Tanga, with a population of over 2 million (16). The highland and upper parts of the area are characterized by urbanization, densely populated rural areas and cultivation. The lowlands have scattered croplands associated with smaller settlements, usually close to the Pangani River. Arid rangelands make up much of the remaining landscape (17). Variations in the local climate in the area are mostly related to topography. The area has two distinct seasons, the rainfall season between March to May, and the dry season during the other months.

The major river in the area is Pangani River which starts as a series of small streams on the southern sides of Mt. Kilimanjaro and Mt. Meru, and flows for about 500 km before draining into the Indian Ocean at Pangani town in Tanga region. Its major tributaries are Kikuletwa and Ruvu Rivers. Others tributaries include Kikavu, Mkomazi and Soni Rivers (18).

Sample collection

Sediment samples were collected from 12 locations along the main Pangani River and five of its major tributaries; Kikuletwa, Kikavu, Ruvu, Mkomazi and Soni as described in Table 1 and indicated in Figure 1.

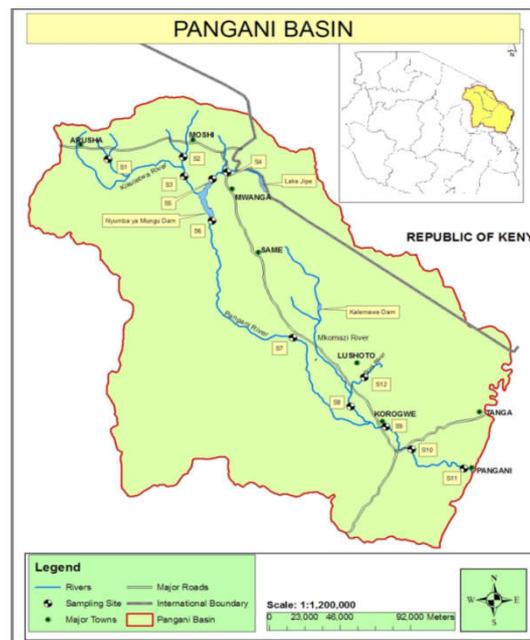


Figure 1. Map of Pangani river basin indicating the sampling locations

Table 1: Sampling locations

Site Code	River/Location	Coordinates	Elevation (AMSL)
S1	Kikuletwa River at Karangai	S 03° 26.44 E 036° 51.27	1020 m
S2	Kikavu River at Kikavu Chini	S 03° 26.22 E 037° 18.05	1019 m
S3	Kikuletwa River at Msitu wa Tembo	S 03° 33.00 E 037° 18.25	741 m
S4	Ruvu River at Kifaru	S 03° 31.35 E 037° 33.42	709 m
S5	Ruvu River at Tingatinga	S 03° 33.17 E 037° 29.09	702 m
S6	Pangani River at Nyumba ya Mungu	S 03° 50.18 E 037° 27.31	669 m
S7	Pangani River at Buiko	S 04° 38.58 E 038° 02.34	534 m
S8	Mkomazi River at Makayo	S 04° 39.35 E 038° 04.36	450 m
S9	Pangani River at Korogwe	S 05° 09.50 E 038° 28.22	287 m
S10	Pangani River at Hale	S 05° 17.37 E 038° 36.06	266 m
S11	Pangani River at Mseko	S 05° 24.26 E 038° 52.04	06 m
S12	Soni River at Soni, Lushoto	S 04° 50.27 E 038° 22.09	1182 m

All samples were collected by grabbing using a stainless steel scoop from the upper 6 cm of the river-bottom sediments. After collection, samples were stored in polyethylene bags and transported to the laboratory at the Department of Chemistry, University of Dar es Salaam, Tanzania, where they were kept frozen at 4 °C before preparation. Samples were later on freeze-dried at 10⁻¹ bar and -57°C. Other portions of the samples were stored in sealed sample vials and transported to the Department of Chemistry, University of Antwerp, Belgium for characterization by micro-Raman spectroscopy.

XRD Analysis

XRD analyses were performed in both bulk sediment samples and separated clay fractions. The clay fractions were separated from the bulk sediments by dry-sieving and decantation experiments using gravity settling of particles in deionised water and centrifugation as per the method described by Verwaal and Mulder (19).

The X-ray diffraction patterns were recorded by an Inxitu BTX-231 Benchtop X-ray Diffraction/Fluorescence spectrometer at the Department of Geology, University of Dar es Salaam in Tanzania. The following conditions were used in the analysis: CoK α , 30 kV, 30 mA, and graphite monochromator. Obtained XRD patterns were compared to standard database using xpowder software loaded with difdata mineral database, published literature and an in-house library for the identification of the different mineralogical species present in the samples.

Micro-Raman Spectrometry measurements

Previously freeze-dried and homogenized sediment samples were ground in an agate mortar and sieved to 63 μ m meshes prior to analysis. A micro-Raman spectrometer (Renishaw in Via Reflex, Wotton under Edge, UK) equipped with two lasers 514 nm and 785 nm was used for the sediment characterization at the Department of Chemistry, University of Antwerp in Belgium. The laser beam was focused on the sample by means of a microscope objective, which also works as a backscattered light collector. The spectra were recorded using a coupled Peltier cooled charge-coupled device (CCD) detector. A colour micro-video camera mounted on the microprobe was used in focusing. A joystick controlled X-Y plate mounted on a tripod was used to set the focus, and the Z axis was manually changed whenever necessary. The instrument was calibrated every working day by using the 520cm⁻¹ Raman shift of a silicon wafer (Figure 2).

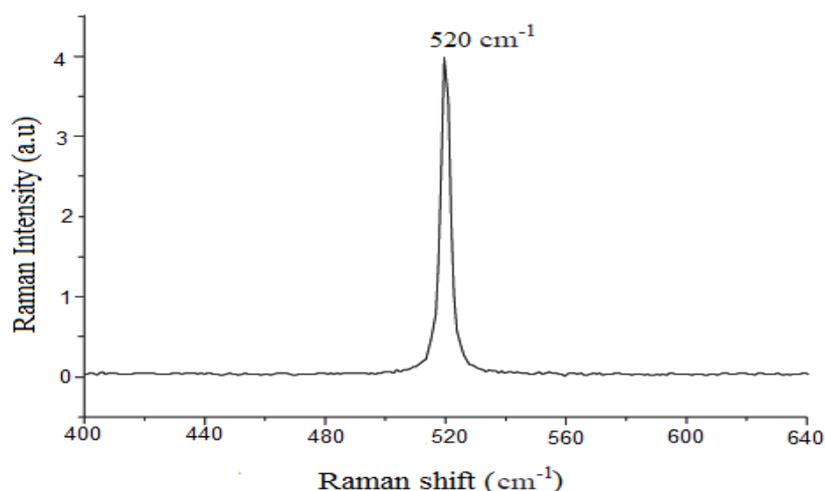


Figure 2. A Raman spectrum of silicon used for instrument calibration

The measurements of the sediments sample involved analysis of individual grain particles as were viewed in the micro-video camera. The set-up measurements such as number of acquisitions, exposure time and laser power were varied to improve the signal-to-noise ratio, but 100 accumulations of 2 s each were used in most of the cases. In the measurements, the microscope objectives were varied between 10x, 50x and rarely 100x. Confocality was also varied between high and standard. The studied interval of wavenumbers was also varied. Analyzed particles' morphology was also noted so as to be able to detect any change which could indicate sample burn-out or decomposition during the measurement.

The measurement process was started by viewing the individual grains through the micro-video camera, selecting the specific one to collect the Raman spectra and storing the image before an exposure to the laser beam. In most cases a quick scan using the Renishaw SynchroScan mode from 100 to 3200 cm^{-1} was performed on samples to detect indications of Raman activities. If some activities were observed, a static mode was then run targeting that specific range. In cases where the stored spectrum had a bad signal-to-noise ratio, the measurement process was then repeated using a different laser power. Whenever necessary, sample bleaching was employed to reduce the background. The measurements are repeated in correspondence of different points to obtain representative results. The basis of species identification by Raman spectroscopy is the comparison of the spectrum of an unknown material with reference spectra (Raman spectral fingerprinting). In this study, spectra obtained from Raman measurements of the sediment samples were compared with those in an in-house library as well as an open source spectra library to allow the precise identification of the minerals residing in the sediments.

RESULTS AND DISCUSSIONS

Mineralogical compositions identified from the XRD analysis

The clay mineral fractions that were separated from the bulk sediment samples mostly contained illite, kaolinite and smectite. From the XRD patterns, the most frequently observed mineral phases in the analyzed sediment samples were the following;

Quartz: SiO_2

Albite: $[\text{Na}(\text{Si}_3\text{Al})\text{O}_8]$ or $[(\text{AlSi}_3)\text{NaO}_8]$

Muscovite: $[\text{K}_{0.92}\text{Na}_{0.01}\text{Al}_{1.87}\text{Mg}_{0.02}\text{Ti}_{0.02}\text{S}]$

Oligoclase: $[(\text{Na}_{0.723}\text{Ca}_{0.277})(\text{Al}_{1.277}\text{Si}_{2.7})\text{O}_4]$

Orthoclase: $[\text{K}(\text{Si}_3\text{Al})\text{O}_8]$,

Zeolite-L: $[\text{K}_6\text{Na}_3(\text{Al}_9\text{Si}_{127})\text{O}_93\text{H}_{102}]$

Sediment samples from all locations showed the presence of quartz. The other most commonly found minerals were albite, zeolite-L and muscovite originating from the continental soils and weathered rocks in and around the area, with muscovite reflecting the influence of metamorphic rocks while zeolites mostly pointing to the influence of volcanic (20). The list of minerals identified from the samples and the sampling site where they were found is given in Table 2 with their literature compositions, while Figure 3(a-d) give some representative X-ray diffractograms of the samples.

Table 2: List of minerals identified in the XRD analysis

Site	Mineral phase found	Composition
S1	Albite Quartz Microcline Wollastonite Orthoclase Muscovite Oligoclase Zeolite-L	Na(Si ₃ Al)O ₈ SiO ₂ (AlSi ₃)(K.89Na.11)O ₈ CaSiO ₃ K(Si ₃ Al)O ₈ K.92Na.01Al1.87Mg6Ti.02S (Na.723Ca.277)(Al1.277Si2.7)O K ₆ Na ₃ (Al ₉ Si ₁₂₇)O ₉₃ H ₁₀₂
S2	Zeolite Ulvospinel Wollastonite Quartz Xenotime	(K,Ba)-G,L K ₂ .19Ba ₆ .68(Al ₁₈ Si ₁₈)O ₁₁₁ Ti(Fe _{1.42} Mn _{5.76})O ₄ CaSiO ₃ SiO ₂ YPO ₄
S3	Wollastonite Albite, Quartz, Vermiculite	CaSiO ₃ Na(Si ₃ Al)O ₈ SiO ₂ (Mg,Fe ²⁺ ,Al) ₃ (Al,Si) ₄ O ₁₀ .4(H ₂ O)
S4	Albite, Quartz	Na(Si ₃ Al)O ₈ SiO ₂
S5	Triphylite Labradolite Albite Oligoclase Andesine	LiMn.089Mg.234Fe.687PO ₄ (Ca.325Na.16(Si1.19Al.81)O ₄ (AlSi ₃)NaO ₈ (Na.723Ca.277)(Al1.277Si2.7)O ₄ Ca.24Na.26(Al.735Si1.265)O ₄],
S6	Clinohypersthene Albite	Mg.636Fe1.332Ca.032Si ₂ O ₆ Na.25(Al.25Ge.75)O ₂
S7	Xenotime Orthoclase Quartz	YPO ₄ KAlSi ₃ O ₈ SiO ₂
S8	Labradolite Albite Oligoclase Andesine Quartz Zeolite	(Ca.325Na.16(Si1.19Al.81)O ₄ (AlSi ₃)NaO ₈ (Na.723Ca.277)(Al1.277Si2.7)O ₄ Ca.24Na.26(Al.735Si1.265)O ₄ SiO ₂ K ₆ Na ₃ (Al ₉ Si ₁₂₇)O ₉₃ H ₁₀₂
S9	Quartz Zeolite Orthoclase Muscovite	SiO ₂ K ₆ Na ₃ (Al ₉ Si ₁₂₇)O ₉₃ H ₁₀₂ KAlSi ₃ O ₈ KAl ₂ (Si ₃ Al)O ₁₀ (OH,F) ₂
S10	Xenotime Quartz	Y(PO ₄) SiO ₂
S11	Quartz Zeolite	SiO ₂ [K ₆ Na ₃ (Al ₉ Si ₁₂₇)O ₉₃ H ₁₀₂]
S12	Quartz Albite Zircon Zeolite	SiO ₂ Na(Si ₃ Al)O ₈ ZrSiO ₄ [K ₂ .19Ba ₆ .68(Al ₁₈ Si ₁₈)O ₁₁₁]

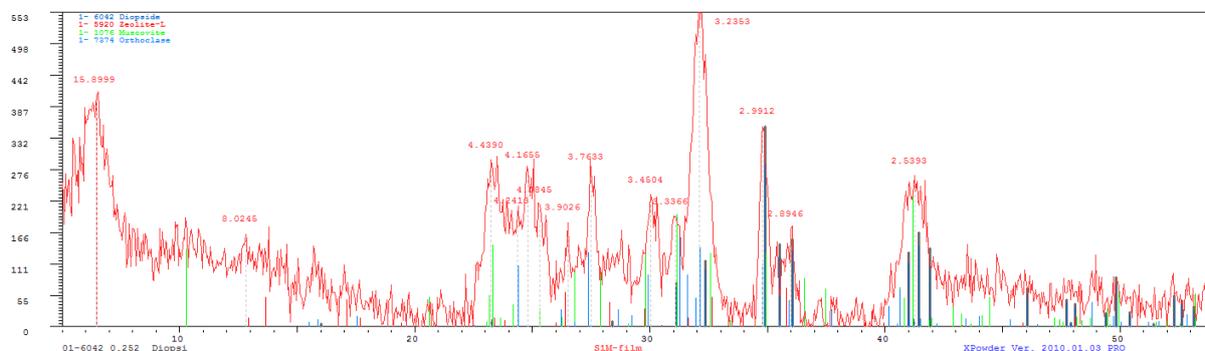


Figure 3(a) XRD diffraction pattern of sediment from site S1

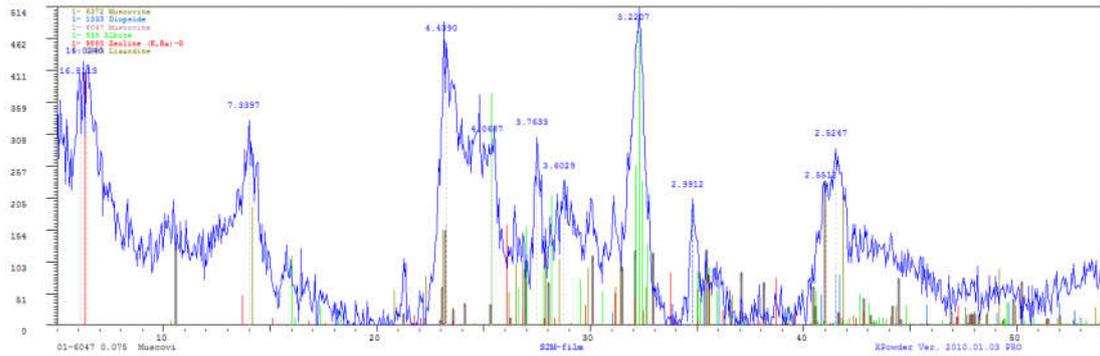


Figure 3(b) XRD diffraction pattern of sediment from site S2

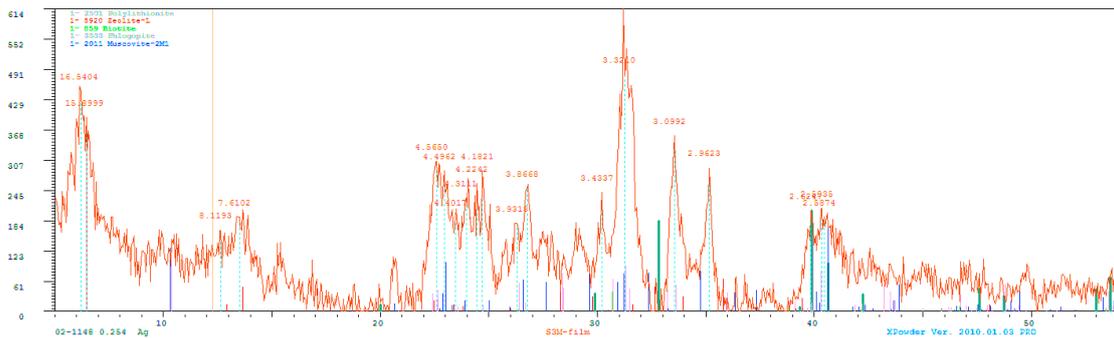


Figure 3(c) XRD diffraction pattern of sediment from site S3

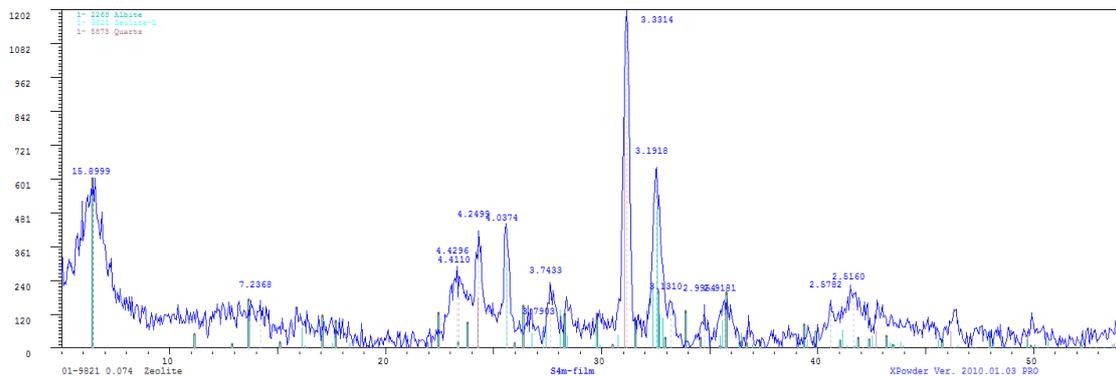


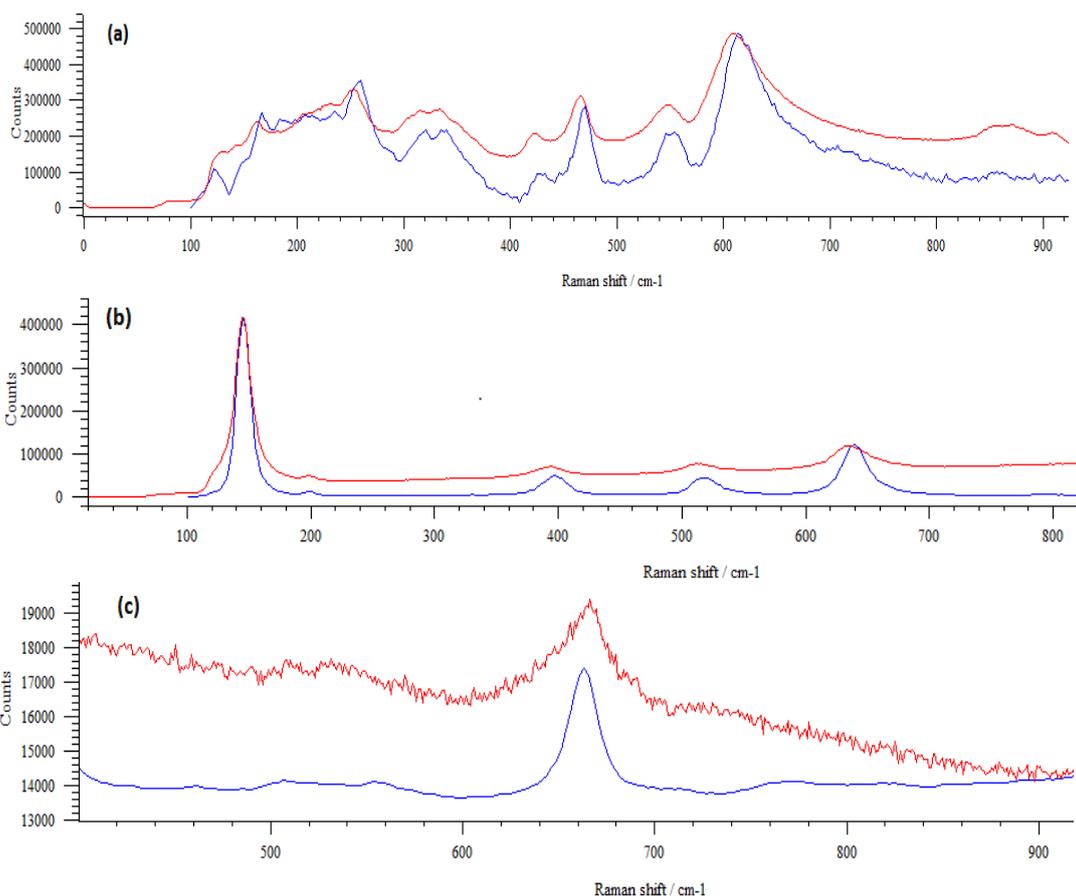
Figure 3(d) XRD diffraction pattern of sediment from site S4

Mineralogical compositions identified from the MRS analysis

Multiple spectra bands were obtained for each sediment sample measured by using the variety of conditions previously described. Figure 4(a-f) shows examples of the Raman spectra obtained from the riverbed sediment samples the twelve locations in this study, matched with the database spectra that were used to identify them. Some spectra were of good quality and could be easily matched with the database with a high degree of certainty for the identified compounds. However, there were also some cases where peaks could not be assigned, and therefore not identified. Table 3 summarizes the compounds that were identified, their chemical formulas from literature, their characteristics Raman bands and the sites where they were recorded. Plates 1(a-d) give examples of individual particles in the sediment grains as were visualized through the micro-video camera of the micro-Raman spectrometer. Generally, the identified minerals reflect the geologic setting of the area. Some of the compounds like quartz, albite and orthoclase were identified by both techniques, while others were identified by only one.

Table 3: List of minerals identified in the MRS measurements

Compound	Chemical formula	Characteristics Raman bands (Wave numbers, cm ⁻¹)	Sites recorded
Quartz	SiO ₂	463	All
Calcite	CaCO ₃	1086, 282, 713	S3, S6, S8, S11
Aragonite	CaCO ₃ (different CO ₃ ²⁻ orientation)	1085, 208, 706	S3, S6, S11
Dolomite	CaMg(CO ₃) ₂	1097, 177, 299, 725	S3
Spessartine	Mn ₃ Al ₂ (SiO ₄) ₃	912, 357, 855	S7, S8, S9, S12
Anatase	TiO ₂	145, 640, 396, 512	S1, S2, S3
Orthoclase	KAlSi ₃ O ₈	509, 473, 273, 1121	S5, S7
Rhodocrosite	MnCO ₃	1087, 291, 179	S6, S11
Richterite	Na ₂ CaMg ₃ Fe ₂ (Si ₈ O ₂₂ (OH) ₂)	679, 230, 1066,	S5
Rutile	TiO ₂	162, 323, 433	S1, S2, S3
Illmenite	Fe ²⁺ TiO ₂	694, 347, 602	S2, S6
Sanidine	(K, Na)(Al, Si) ₄ O ₈	512, 159, 282	S6, S7
Biotite	K(Mg, Fe ²⁺) ₃ [AlSi ₃ O ₁₀ (OH, F) ₂]	618, 470, 258, 340, 549	S1
Augite	(Ca, Na)(Mg, Fe, Al)(Si, Al) ₂ O ₆	663, 1006	S2, S6
Kaolinite	Al ₂ Si ₂ O ₅ (OH) ₄	1085, 153, 537	S6
Lepidocrosite	FeO(OH)	244, 1295, 386	S9
Sphene	CaTiSiO ₅	149, 613, 251	S1
Albite	NaAlSi ₃ O ₈	511, 154, 279	S4, S9, S11
Pyroxene	(Ca, Na)(Mg, Fe, Al)(Al, Si) ₂ O ₆	663	S6
Pyrolusite	MnO ₂	610	S1, S2, S9, S12



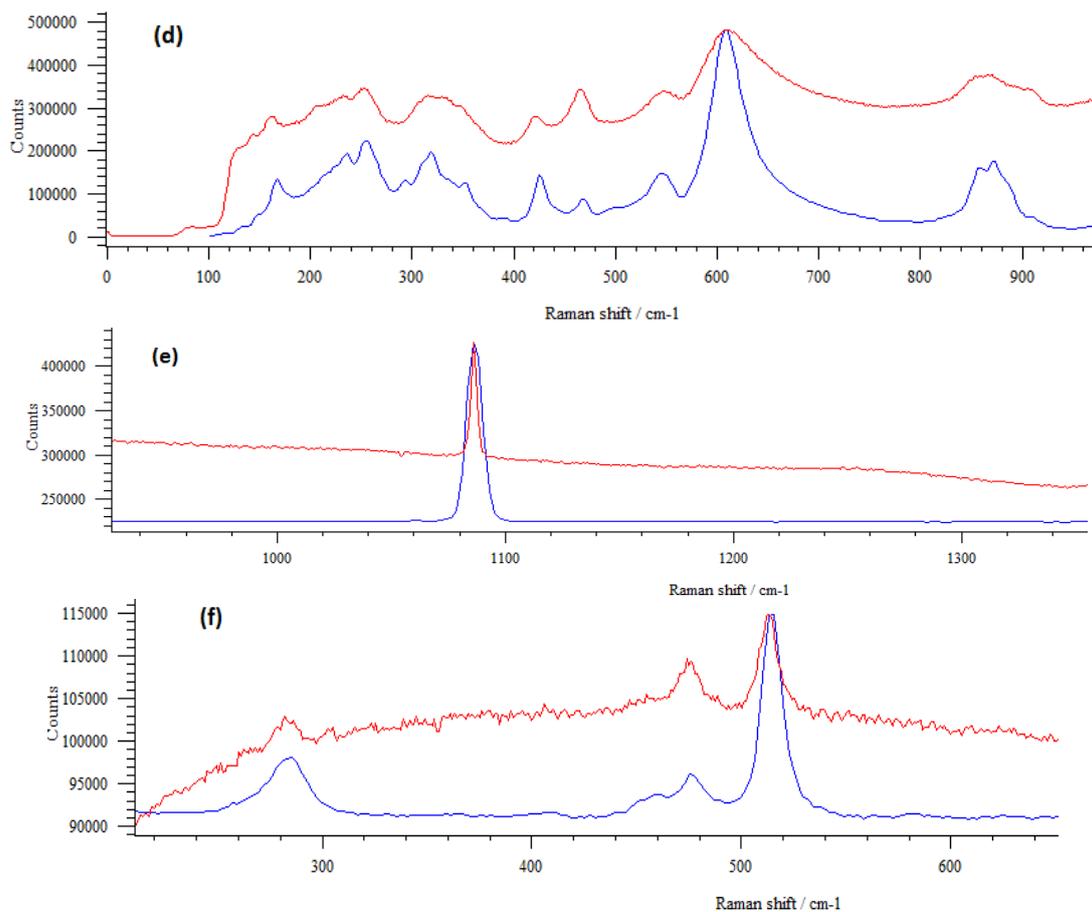
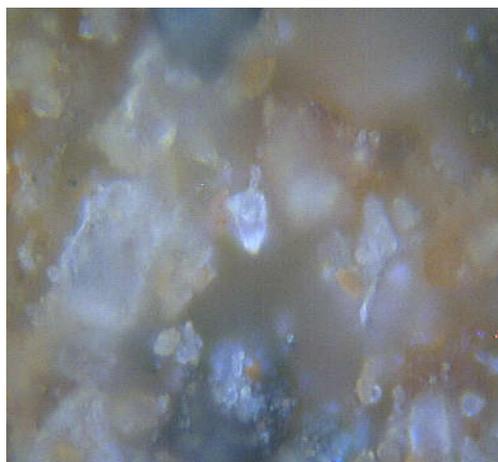


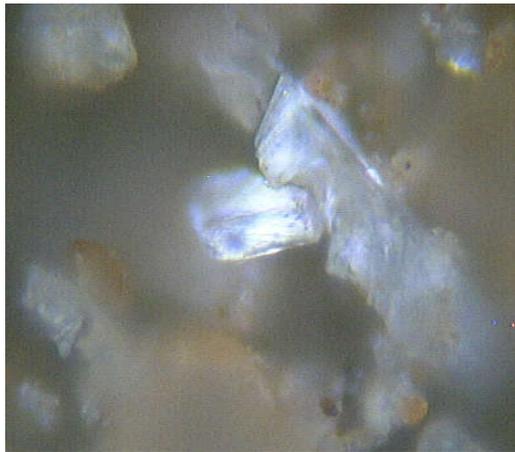
Figure 4. Examples of micro-Raman spectra used for mineral identification, with reference spectra shown in blue and samples in red **(a)** Biotite in sample S1 **(b)** Anatase in sample S1 **(c)** Pyroxene in sample S6 **(d)** Sphene in sample S1 **(e)** Aragonite in sample S6 **(f)** Orthoclase in sample S7



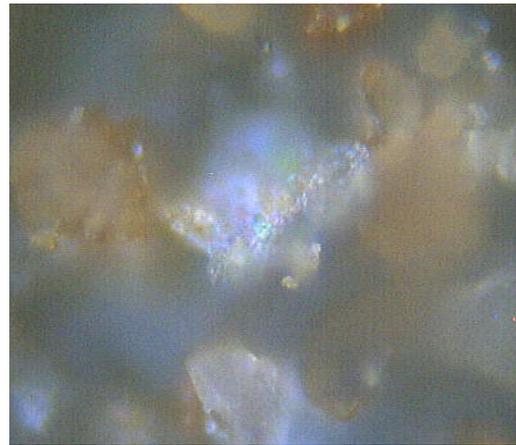
(a) Quartz, visualized in S4



(b) Sanidine, visualized in S6



(c) Spessartine, visualized in S8



(d) Albite, visualized in S7

Plate 1. Examples of visualization of some of the identified mineral grains in the analyzed sediment samples

CONCLUSIONS

Mineral compositions of sediment samples from the Pangani river basin have been preliminary characterized. Illite, kaolinite and smectite were the clay minerals identified. The MRS measurements also identified spessartine, calcite and pyrolusite in more locations than most of the other identified minerals. The technique of micro-Raman spectroscopy has proved to be applicable in the study of mineral phases of the Pangani river basin sediments in that, the minerals could be easily identified by their Raman Spectra. Since the measured Raman signals are often characteristic for analyzed compounds, Raman spectroscopy allowed for identification of very complex compounds, which could otherwise not be done by other techniques. This investigation shows that riverbed sediments from the location are composed of a wide range of mineral phases from the rich geologic diversity of the area. A more detailed study is therefore suggested for more comprehensive insight. However, due to its preliminary nature, this study could not establish the geochemical signature of sediments from individual locations such as the specific spatial distribution patterns of the mineral assemblages and their relation to the source rocks. In future, quantitative analyses could be used for more accurate mineral analysis and to improve the knowledge on geochemical characteristics of sediments from this important regional resource.

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