Report to SAHRA on Middle Stone Age lithic raw material sourcing from Olieboomspoort (Limpopo Province, South Africa)

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This study forms the core of the Master dissertation submitted in 2022 to the Faculty of Science at the University of the Witwatersrand by Dineo Masia. The dissertation is attached as an additional file to this heritage report. It contains extensive tables, figures and photographs of the material analyzed. References mentioned here are also listed in the bibliography of the Master dissertation. Here, we have extracted the information directly relevant to the study of the lithic artefacts from Olieboomspoort and for which a permit was granted by SAHRA in 2021.

MATERIAL

A selection of 65 lithics from OBP was analysed. These lithics were selected from the A. Val (2018-2019) collection. In the selected OBP material, quartzite makes up 43.1% of the lithic assemblage and is followed by shale (30.8%), dolerite (12.3%), sandstone (9.2%), quartz (3.1%), and chert raw materials (1.5%). These lithics are flakes and fragments that were selected because they are not technologically and typologically important and they cover a variety of different rock types. Therefore, they can be analysed with destructive techniques. Within the OBP collection, lithics from GS décapages 1, 4, 10, 13, 14, and 16, as well as those from YRS décapages 1, 7, and 15, were analysed.

Lithics were accessed at the Leswika Archaeology Laboratory at the Evolutionary Studies Institute (University of the Witwatersrand). Images of representative samples of the lithics (based on rock types) from both sites are shown in Figure 1. Descriptions and pictures of all the lithics can be found in APPENDIX A of the Master dissertation of Dineo Masia. Permit (ID: 3281) from the South African Heritage Resources Agency were received for the destructive analyses that were conducted on a few carefully selected lithics (unretouched flakes).

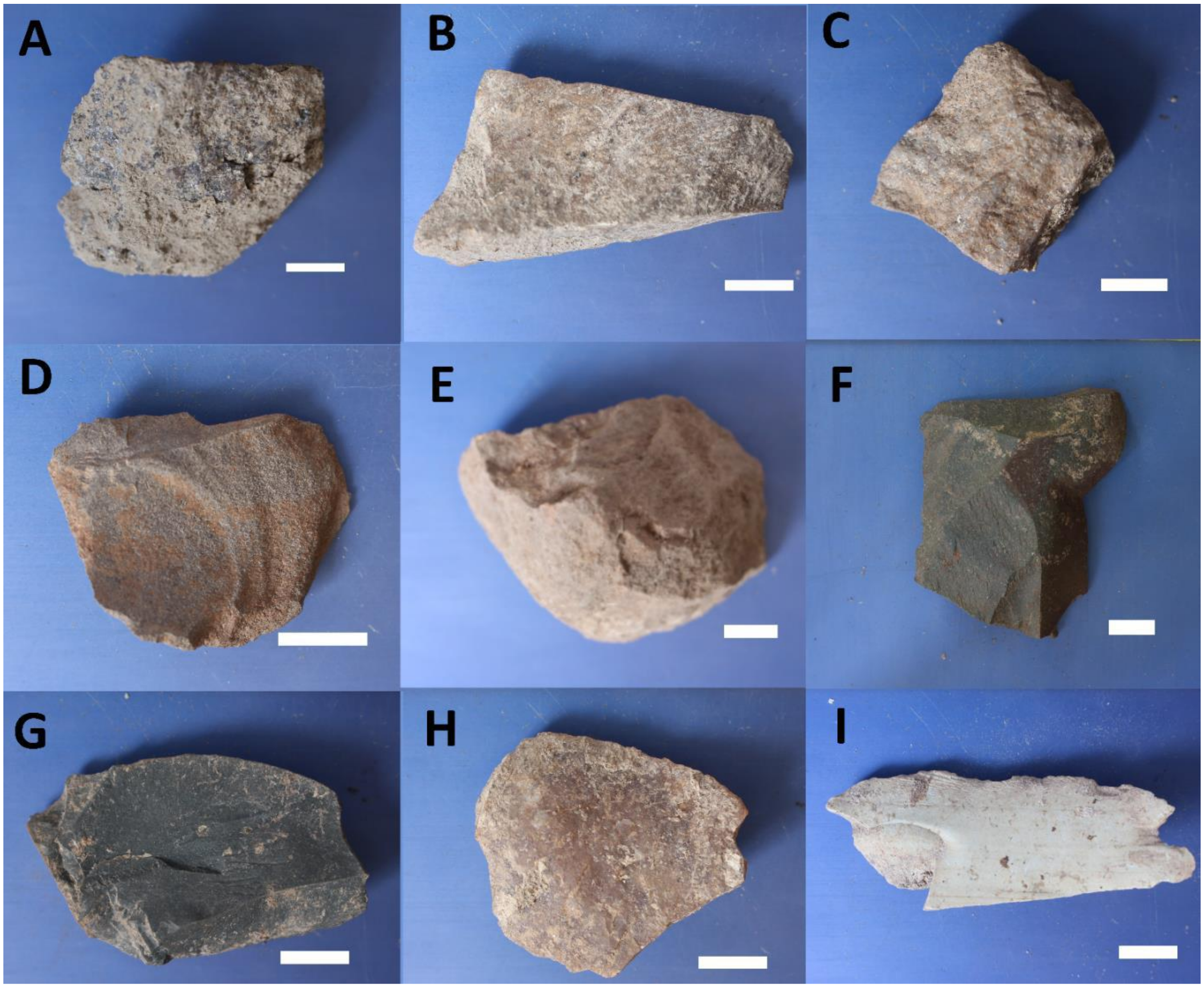


Figure 1: Representative samples of OBP lithics. A) No. 2402 (sandstone), B) No. 1194 (sandstone), C) No. 486 (quartzite), D) No. 458 (dolerite), E) No. 3478 (quartzite), F) YRS 1-D (shale), G) No. 164 (shale), H) No. 493 (quartz), and I) No. 412 (chert). These are all unretouched flakes. The white bar represents 1 cm.

Geological materials

The areas chosen for prospection surveys around OBP are based on the rock types of the representative samples that were selected for this study from the A. Val (2018-2019) lithic collections. This was done to link lithics to possible outcrops. Promising locations were identified using geological literature, the 2326 Ellisras Basin and 2428 Nylstroom maps (Geological Survey (South Africa)), and satellite images of the areas around the sites (Google Earth). Based on the rock types noted in the lithic assemblage and geological research, prospection areas of a 12 km radius around both sites were expected to be sufficient for geological sampling although this was not the case around OBP because of flat areas without outcrops and a lack of access to some private properties. Geological samples were taken from both primary and secondary contexts at OBP. The primary contexts are *in situ* outcrops. The secondary contexts are samples like pebbles collected along the road or river, and *ex situ* samples with the same lithology as outcrops surrounding the sites.

At OBP, preliminary assessments show that the selected lithic assemblage consists of quartzite, shale, sandstone, dolerite, and quartz. These rock types (except quartzite) are expected in the Mogalakwena Formation based on geological literature (Corcoran *et al*., 2013; Geological Survey (South Africa)). Geological samples representing these rock types were collected within 30 km SSE of the site and within about 13 km NNW of OBP in the Mogalakwena Formation and the Cleremont Formation. The geological assemblage consists of 15 quartzite samples, two shale samples, five sandstone samples, four dolerite samples, and 22 quartz samples. Most of the geological samples were collected in secondary context (89.7%; *ex situ*) as pebbles and cobbles along the Rietspruit River, roads, and on a mountain at the Zandrivier Farm although some were sampled from primary context (10.3%; *in situ*) outcrops. This was due to the lack of *in situ* outcrops in the area around OBP. Samples from *in situ* outcrops were photographed and then removed with a geological hammer. These were then labelled and their geographical coordinates were recorded using the Garmin eTrex® GPS.

Each potential source is described as a 'locality' in Table 1. A collection of 48 geological samples from 17 localities was collected around OBP (Table 1). During the prospection, general descriptions (APPENDIX A) of the outcrops were recorded and images were taken for reference.

Table 1: The number of samples collected at each stop, their rock types and the distance at which they were collected.

OBP Localities	**Rock Types**	**Context**	**No. of Outcrop samples**	**Distance from site**
MC1	Quartz conglomerate	Primary	**1**	28-29 km
MC2	Quartz conglomerate	Primary	**1**	28-29 km
MC3	Quartz conglomerate	Primary	**1**	28-29 km
CMF1	Quartzite	Primary	**1**	14.5 km
CMF2	Quartzite	Primary	**1**	14.5 km
BPC1	Quartzite, quartz	Secondary	**6**	28-29 km
BB 1	Quartz conglomerate	Primary	**1**	28-29 km
OBP 1	Quartz and quartzite conglomerate	Secondary	**1**	130 m
OBP 2	Quartz conglomerate	Primary	**1**	615 m
OBP 3	Quartz, sandstone	Primary	**3**	615 m
OBP 4	Quartzite, quartz, dolerite	Secondary	**7**	175 m
OBP 5	Quartzite, quartz, dolerite	Secondary	**6**	9 km
OBP 6	Quartzite, quartz	Secondary	**6**	485 m
OBP 7	Sandstone	Secondary	**1**	485 m
OBP 8	Sandstone	Primary	**3**	155 m
OBP 9	Quartz	Secondary	**2**	13-14 km
OBP 10	Quartzite, quartz, shale/meta-sedimentary	Secondary	**6**	13-14 km

METHODS

Macroscopic (including stereomicroscopic) analysis

Stereomicroscopic analysis of the samples was conducted with the use of an Olympus SZX16 stereomicroscope (magnification range: 0.7x -11.5x) at the Leswika Archaeology Laboratory (Evolutionary Studies Institute, University of the Witwatersrand). Petrological features that aid in rock identification and could help form general rock groups were studied. These include:

- The colour of the rock
- Lustre
- Inclusions and foliations
- Grain size
- Grain shape
- Intergranular relationships (interlocking, disseminated, etc.)
- Nature of the cortex was analysed to establish the contexts of the samples (primary or secondary)

Portable X-ray Fluorescence Spectrometry

Portable X-ray Fluorescence Spectrometry (pXRF) spectrometry is an analysis that quantifies the concentration of elements in a sample. It is used in this project to identify and group samples according to rock types. From these results, preliminary information about lithics and their geological sources is provided.

This method does not require much sample preparation and it produces elemental compositions relatively fast. Not only that, but it is also inexpensive and very easy to use. A few drawbacks of pXRF spectrometry are that it is unable to register light elements like Na and has low sensitivity. These drawbacks could lead to the misidentification of a rock. However, pXRF spectrometry remains a good non-destructive method (Shackley, 2011). Especially in a study such as this one because archaeological samples are being analysed and it is a priority not to destroy them. For that reason, pXRF is used to see if it can link lithics to geological samples.

Samples were analysed with no preparation using the Olympus Vanta™ pXRF instrument (40 kV beam) at the Leswika Archaeology Laboratory. The instrument is industrial and was commercially calibrated (factory calibration). The instrument was considered reliable because it provided results that were supported by ICP-MS analysis. I am, however, aware that this is not an absolute measure of accuracy. PXRF errors were provided for each analysed element although I am not certain how they were calculated since this an industrial instrument. I have included them in Appendix B (Table B3 and Table B4). Smaller lithics that could fit in the instrument were analysed while it was fixed on a stand. The larger lithics and outcrop samples that could not fit into the instrument underwent hand-held analyses. Each sample was analysed three times (45 second reading for each analysis: 15 seconds for the minor elements and 30 seconds for major elements). The beam from the pXRF instrument travelled through the air before penetrating the rock because the analyses were not conducted in a vacuum/ controlled environment. The following elements with concentrations generally above the level of detection were recorded: Al, Si, P, S, K, Ca, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, As, Rb, Sr, Y, Zr, Nb, Mo, Pb, and Th.

Principal Component Analysis

Principal Component Analysis is an unsupervised, multivariate statistical analysis. It reduces the number of variables in a dataset and maximiases variability within that dataset (Mauran *et al*., 2022).

PCA was conducted on the raw data obtained through pXRF analysis. Columns from the raw pXRF data in which more than 10% of the samples showed elemental amounts below the level of detection (LoD) were deleted. The LOD readings in the remaining columns were replaced with a constant number that is half of the minimum reading for the element. From there, PCA code (Mauran *et al*., 2022; Baxter, 1994)), and the packages ggplot2, ade4, MASS, MVN, and plyr, were used in R Studio to produce the plots to differentiate formations and to link lithics to these formations.

Thin sections petrography

Thin section analysis provides the mineral compositions and estimates of mineral proportions in a sample. It offers the most accurate petrographic information in primary analyses (Andrefsky, 1998). This is important because mineral modal abundance estimates are pivotal in rock analysis and identification (Chaves, 1949).

The petrography of the lithics and geological samples was analysed by looking at features such as:

- Granular nature (morphology, sizes, relationships)
- Textures (trachytic, ophitic, foliations)
- Extinction angles
- Colour

These are features used to identify and group rocks. Quartzite samples were identified and compared by the presence of undulose extinction and Bohm lamellae in addition to the abovementioned features. The presence of interlocking grains, stylolites and syntaxial cement; as well as the shape of grain boundaries (convex-concave), was also used to describe quartzites.

From the studied sample set, 21 samples (12 lithics and nine geological samples, see Table 3.3) were cut in half with a rock cutter at the School of Geosciences (University of the Witwatersrand) for thin sections and ICP-MS powders.

To make the thin sections, the rocks were cut using a diamond saw; embedded in resin, ground flat with corundum 600 and 1200 and water. These were then polished using a polishing machine. The representative sample of OBP includes the following rock types: dolerite/andesite, shale/meta-sedimentary rocks, white quartzite, black quartzite, red quartzite and quartz. The rock types analysed are those represented in both the lithic assemblage and the geological assemblage. Chert and quartz samples were not analysed through thin section analysis because no grains would be visible using a microscope. As mentioned, three different types of quartzites (black, white, and red) were selected for the representative sample for grouping, however, red quartzite geologic samples were not found during the prospection survey.

Inductively Coupled Plasma Mass Spectrometry

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) is a highly sensitive element detection analysis. It is also used to quantify elemental concentrations. ICP-MS provides more refined, and accurate, elemental results in comparison to pXRF spectrometry. The error limits for the elements analysed were not provided with the results because the analysis was conducted in the EarthLab (University of the Witwatersrand). This is the final analysis in this project used to identify rocks and link lithics to geological samples.

ICP-MS is reliable because it can detect elements at low detection limits. It also detects multi-element isotopes at high spectral resolutions (Thomas, 2013). This analysis is preferable because, unlike pXRF spectrometry, it can quanitify and detect a larger range of elements, including lighter elements like Na (Thomas, 2013).

As shown in Table 2, 13 samples from OBP were analysed using ICP-MS. The samples were prepared using a rock crusher and miller at the School of Geosciences (University of the Witwatersrand) and the powders were sent to the EarthLab (University of the Witwatersrand). They were analysed using a Thermo Scientific iCAP RQ. The Calibration Standards were made at 10, 30, 50, 75 and 100ppb with elements purchased from International Certified Reference Materials (Li, P, Sc, Ti, V, Cr, Co, Ni, Cu, Zn, Ga, Rb, Sr, Y, Zr, Nb, Sn, Sb, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Tl, Pb, U, and Th). From this method, trace element and rare-earth element concentrations were obtained.

Thin section and inductive coupled Plasma Mass Spectrometry Analytical Strategy

Only a few representative samples were chosen, based on rock groups that were established through macroscopic analysis, to undergo thin section and ICP-MS analysis to minimise damage to archaeological material (Table 3 and Table 4). In this table, the localities are given numbers and the samples from each locality are identified using the alphabet (i.e. OBP 8 is the locality and A, B, and C are the samples from that locality).

Table 2: Number of lithics and geological samples that underwent each analytical method at OBP.

OBP		
Analytical method	**Lithics**	**Geological samples**
Macroscopic Analysis	65	48
pXRF	65	48

All samples in this project (lithic and geological) were macroscopically and stereomicroscopically analysed. Then they were analysed through pXRF. PXRF was prioritised before making thin sections and performing ICP-MS analyses because it is non-destructive, and it is fast and easy to use on all samples, providing quick preliminary results.

Table 3: Number of samples, from each rock type, that underwent destructive analyses. The second and third columns show the total number of lithics and outcrop samples in each rock type from the assemblages of both sites. The fourth and fifth columns show the number of lithics and geological samples that underwent destructive analyses in each rock type (i.e. of the eight dolerite lithics at OBP, 2 underwent destructive analyses). *Chert and altered samples were not analysed further than macroscopic analyses they do not occur in both lithic and outcrop assemblages.

Olieboomspoort Rock Shelter				
Total		**Destructive Analyses**		
Rock Type	**Lithics**	**Outcrops**	**Lithics**	**Outcrops**
Dolerite	8	4	2	1
Quartz	2	22	0	0
Quartzite	28	15	2	1
Shale/meta-sedimentary	20	2	2	1
Sandstone	6	5	2	2
*Chert	1	0	0	0
Total analysed	65	48	8	5

Table 4: OBP samples analysed through thin section and ICP-MS analyses, and the rock types that they are made of.

OBP Analysed Geological Samples			
Sample No.	**Rock/Mineral type**	**Analysed through Thin section petrography**	**Analysed through ICP-MS**
CMF1	White quartzite	X	X
OBP 4G	Dolerite	X	X
OBP 7	Black sandstone	X	X
OBP 8A	Black sandstone (with red layers)	X	X
OBP 10A	Shale/meta-sedimentary	X	X
OBP Analysed Lithic Samples			
Sample No.	**Rock/Mineral type**	**Analysed through Thin section petrography**	**Analysed through ICP-MS**
No. 164	Shale/meta-sedimentary	X	X
No. 486	White quartzite	X	X
No. 488	White quartzite	X	
No. 1194	Sandstone	X	X
No. 2402	Black sandstone	X	X
No. 3478	Red quartzite	X	X
No. 458	Dolerite	X	X
YRS 1-D	Shale	X	X

RESULTS

Macroscopic results

The following sections present macroscopic results for each rock type at OBP. Detailed macroscopic descriptions of OBP samples are found in APPENDIX A (Master dissertation of Dineo Masia).

OBP and nearby localities' macroscopic rock descriptions

Quartzite

Quartzite is the most abundant raw material used for lithics at OBP (n=28). The lithic quartzite from the site, although noted as different colours (white, red, and black), is generally represented by similar macroscopic characteristics such as rounded to sub-angular grains that range from fine-grained (0.1 mm) to coarse-grained (4 mm). The quartzite lithic samples are well-sorted and have annealed or interlocking quartz grains (e.g. No. 143, No. 468; No. 488; No. 2374; No. 3474, No. 2332; No. 2364; No. 2395; No. 1795; YRS1-C; YRS1-E) with minimal visible matrix (roughly 25% based on visual comparison to the amount of grains present in the sample). Generally, the texture of the samples is rough and they have a dull lustre although some annealed samples have a smooth, glassy texture. Because of these common characteristics, they are considered as being part of the same group of quartzite. Six samples from the OBP assemblage (No. 488; No. 1195; No. 488; No. 143; No. 3474; and No. 3478) present a visible cortex (Figure 1 A and B). These samples have a brown, orange, and black predominantly dull cortex. These are either smooth or rough. There is no evidence of sedimentary structures in the observed samples.

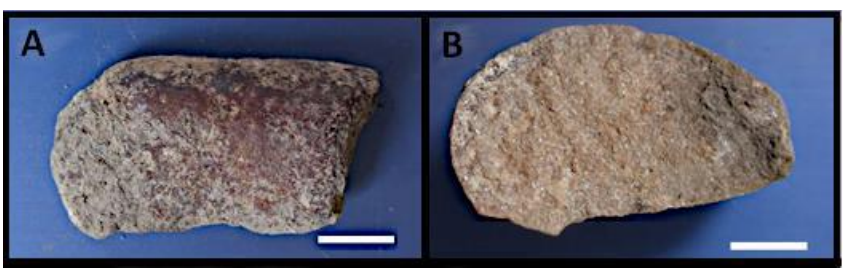


Figure 1: A) No. 1195 shows a smooth, red cortical surface. B) No.488 is an image of the white quartzite at OBP. It is also a sample with a cortex. The white bar represents 1 cm.

The reference quartzite collected around OBP is white, black, or red in colour. Two types of quartzite are noted around OBP in the Mogalakwena and the Cleremont Formations. The general characteristics of quartzite from the Mogalakwena Formation include medium to coarse (>3 mm) quartz grains that are sub-rounded to sub-angular and are generally well-sorted. These grains are usually either white, pink-white, or black. This quartzite has a smooth, glassy fresh surface and the grains are interlocked. The quartzite samples from the Cleremont Formation are similar to those from the Mogalakwena Formation although matrix is noted in samples from localities CMF1 and CMF2 (10%). Quartzite pebbles from both the Mogalakwena Formation and the Cleremont Formation have orange, brown, or pinkish brown cortices. These are usually smooth and have a dull lustre. No sedimentary structures are noted in the collected samples.

Shale

Shales are the second most abundant rock type used at this site (n=20, see Table 3). Three shale groups are noted at OBP. The shales in the general group are mostly dark coloured (black, red-black, brown) and have a smooth surface and dull lustre. These shale lithics are very fine silt-grained (not visible to the bare eye (Figure 2 A)). The second shale group is also dark coloured (brown, black, and grey) and fine-grained although these samples are foliated (e.g. No. 142: Figure 2 B). The third group is similar to the previous two groups in terms of colour and grain size, but white, black, or red grain inclusions are present (e.g. No. 106 A2; Figure 2 C).

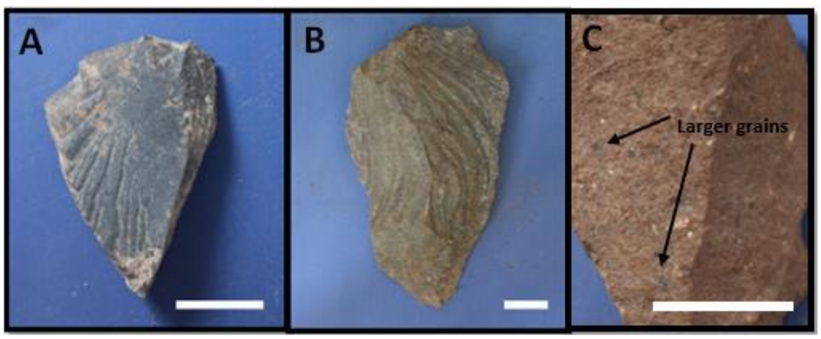


Figure 2: A) No. 143 showing fine-grained texture, B) No. 4677 showing foliation (mica minerals are noted along the lighter foliations), and C) No. 106A2 showing larger grain inclusions. The white bar represents 1 cm.

Only one shale locality was noted around OBP. The shales sampled from OBP 10 are very fine-grained (grains cannot be seen with the naked eye) and black in colour. They are smooth to touch and have a dull lustre. OBP 10B has a foliation noted on its edges.

Sandstone

Generally, two sandstone groups are noted in the OBP lithic assemblage. The first is the general group and the second branches off the general group due to the presence of quartz phenoclasts (No. 2402; Figure 4.3 A, and No. 1194). The sandstone lithics (n=6, see Table 4) in both groups are light brown, red, and/or black in colour. They have rough textures and dull lustres (No. 1286; Figure 3 B) with the exception of No. 1194 (smooth texture) and No. 2366 (glassy lustre Figure 3 C). The quartz grains in the OBP lithic sandstones range from medium to coarse (0.4-1.5 mm) and they show a range of grain shapes (angular to sub-rounded). The quartz grains are well-sorted, and the matrix percentage is low (≤10%). Sedimentary structures are not visible due to the small size of the lithics.

Figure 3: A) No. 2402 showing a coarse-grained texture and quartz phenoclasts. B) No. 1286 and C) No. 2366 are examples of the general characteristics of OBP sandstone lithics. The white bar represents 1 cm.

Samples OBP 3C, OBP 7, OBP 8A, OBP 8B, and OBP 8C are sandstones collected around the shelter (OBP 8 is a large outcrop and the divisions are based on the layers). Three sandstone groups are noted around OBP. Generally, all the sandstones are red or black. They have a rough and dull brown weathered surface with a glassy fresh surface. The grains are coarse (0.7-5 mm), sub-rounded to sub-angular, and they are well-sorted. A group is formed by OBP 3C and OBP 8A because they are banded and have fine oxidised layers (grains: 0.5 mm). OBP 8B and OBP 8C form another group because they are friable sandstones (sandstones sensu stricto). Matrix is only clearly noted in OBP 8C.

Dolerite

No. 182; No. 1241; No. 130; No. 408; No. 458; No. 489; No. 4672; and No. 4688 (n=8, see Table 4) are dolerites. These are aphanitic to medium-grained and have scattered white plagioclase laths (up to 1 mm long) in black matrices (Figure 4). They have a smooth texture and a dull lustre.

Figure 4: No. 1241 shows the medium-grained texture of OBP dolerite samples and fine plagioclase laths. This sample is coated in calcium carbonate. The white bar represents 1 cm.

Ex situ dolerite around OBP is aphanitic and has a brown weathered surface and a black, fresh surface (OBP 4G; OBP 5A, D and, E). Plagioclase laths are only visible in OBP 5D (~10%).

Quartz

No. 493 (pink) and No. 1290 (pink-white) are the only two quartz (monocrystalline) samples noted in OBP lithics (Figure 5). They both have smooth fresh surfaces although No. 493 has a dull lustre and No. 1290 has a glassy one.

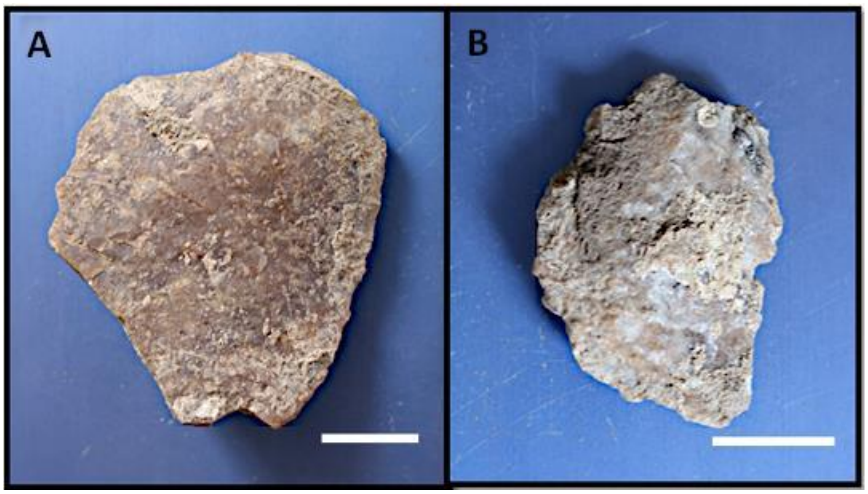


Figure 5: A) No. 493 and B) No.1290 showing their colours, surface textures and lustres. The white bar represents 1 cm.

Most quartz samples are generally pink-white (like the pebbles) but vein quartz, like that sampled at OBP 4D, is pure white. Unlike quartzite pebbles, quartz pebbles have a more pitted and striated cortical surface.

Chert

Very fine-grained, cream white chert (No. 412) also forms part of the OBP lithic assemblage (Figure 6). This sample will not be analysed through any other methods because no chert sources could be sampled near OBP.

Figure 6: Image of the only chert in the OBP assemblage, No. 412. The white bar represents 1 cm.

Portable X-ray Fluorescence data

Olieboomspoort Rock Shelter Scatterplots

Quartzite

After screening several reference materials scatterplots, it appeared that scatterplots Ti/Fe and Si/Al were the most useful to discriminate the reference outcrops. Samples collected from secondary contexts nearby Cleremont Formation all have an Fe content under 1.0% and a Ti content over 700 ppm (Figure 4.12 A). Samples collected from secondary contexts nearby Mogalakwena Formation appear to be more diverse but present higher Fe content. Two localities (OBP 1 and OBP 4) are very different from the rest of the samples collected nearby the Mogalakwena Formation because they have higher Fe content than the rest. From the Si/Al scatterplot (Figure 4.12 B) it is evident that all samples close to the Cleremont Formation have an Al content above 1.1% and an Si content between 32.0% and 44.6%. The *in situ* Cleremont Formation outcrop samples, in particular, have a higher Al content than the secondary samples. Similar to the Ti/Fe scatterplot, the Si/Al scatterplot shows that samples collected from secondary contexts near the Mogalakwena Formation are more diverse than those near the Cleremont Formation. They present higher Al content and lower Si content in comparison to the Cleremont Formation samples. It is observed that some Mogalakwena Formation and Cleremont Formation samples overlap (OBP 6B and BPC1F, and CMF1, OBP 4E, and OBP 6A). Among these overlapping geological samples, CMF1 is the only *in situ* quartzite.

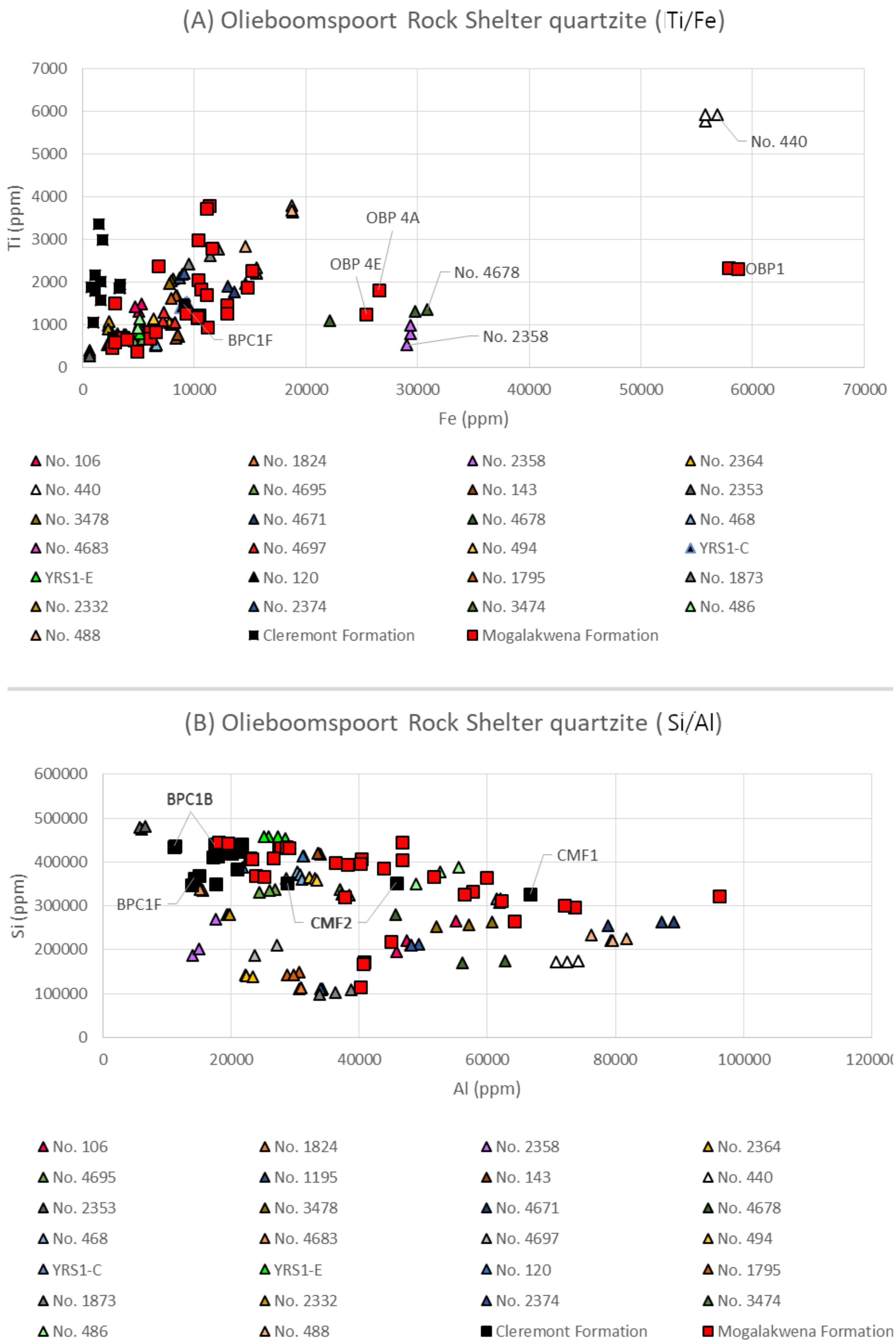


Figure 4.12: A) Ti/Fe scatterplot of quartzite samples at OBP. B) Si/Al scatterplot of quartzite samples at OBP.

Shale

Scatterplot K/Al (Figure 4.13) shows that shale samples collected in secondary context around the Mogalakwena Formation have an Al content between 1.9% and 11.0%. The K content of these samples is below 9500 ppm. Secondary sample OBP 10B has an Al content that is significantly higher than that of OBP 10A.

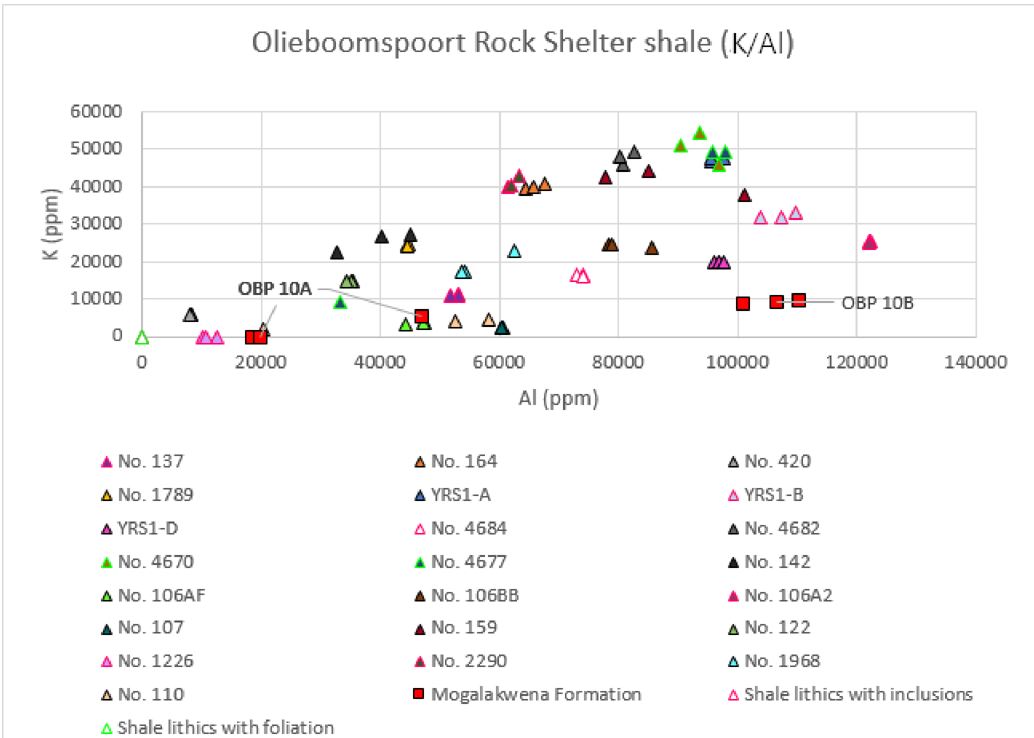


Figure 4.13: A) K/Al scatterplot showing shale samples at OBP.

Sandstone

The most useful scatterplots to discriminate OBP sandstone samples were Ti/Fe (Figure 4.14 A), and Si/Al (Figure 4.14 B). The secondary context sandstone samples collected around the Mogalakwena Formation have a Fe content below 3% and a Ti content below 2400 ppm. Their Al content lies between 2.8% and 7.6% while their Si content is over 18.6%.

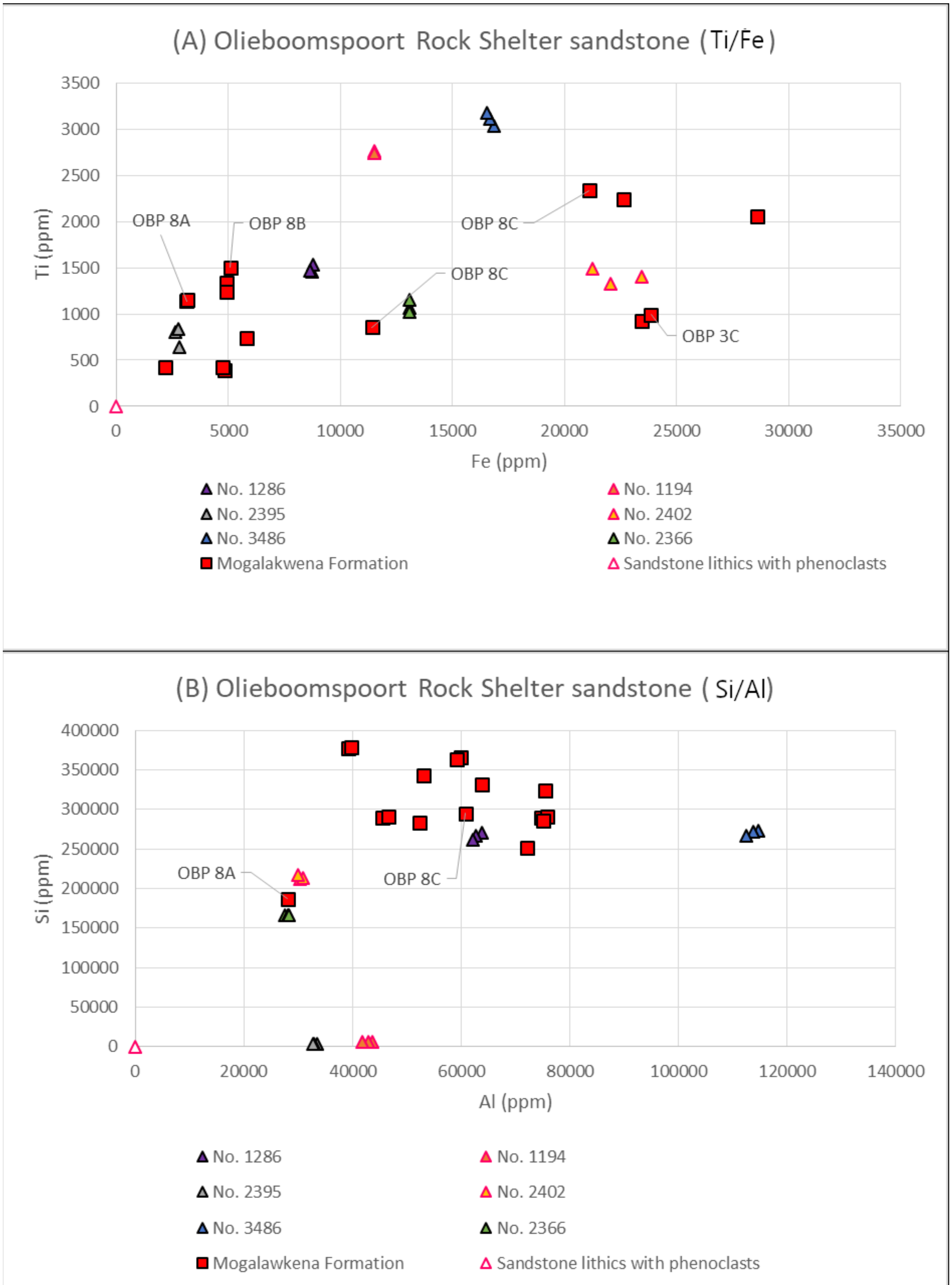


Figure 4.14: A) Ti/Fe scatterplot of sandstone samples at OBP. B) Si/Al scatterplot of sandstone samples at OBP.

Dolerite

Scatterplots Ti/Fe (Figure 4.15 A) and Si/Al (Figure 4.15 B) are the most informative when it comes to comparing OBP dolerite samples. The reference samples collected in secondary contexts around the Mogalakwena Formation have a Fe content between 12.1% and 15.9% and a Ti content between 1.5% and 1.8%. The Al content of these samples is between 7.4% and 12.5%, and the Si content is between 20.2% and 28.7%. *Ex situ* sample OBP 5A plots outside of the Ti range (1.1%) and the Si range (16.5%).

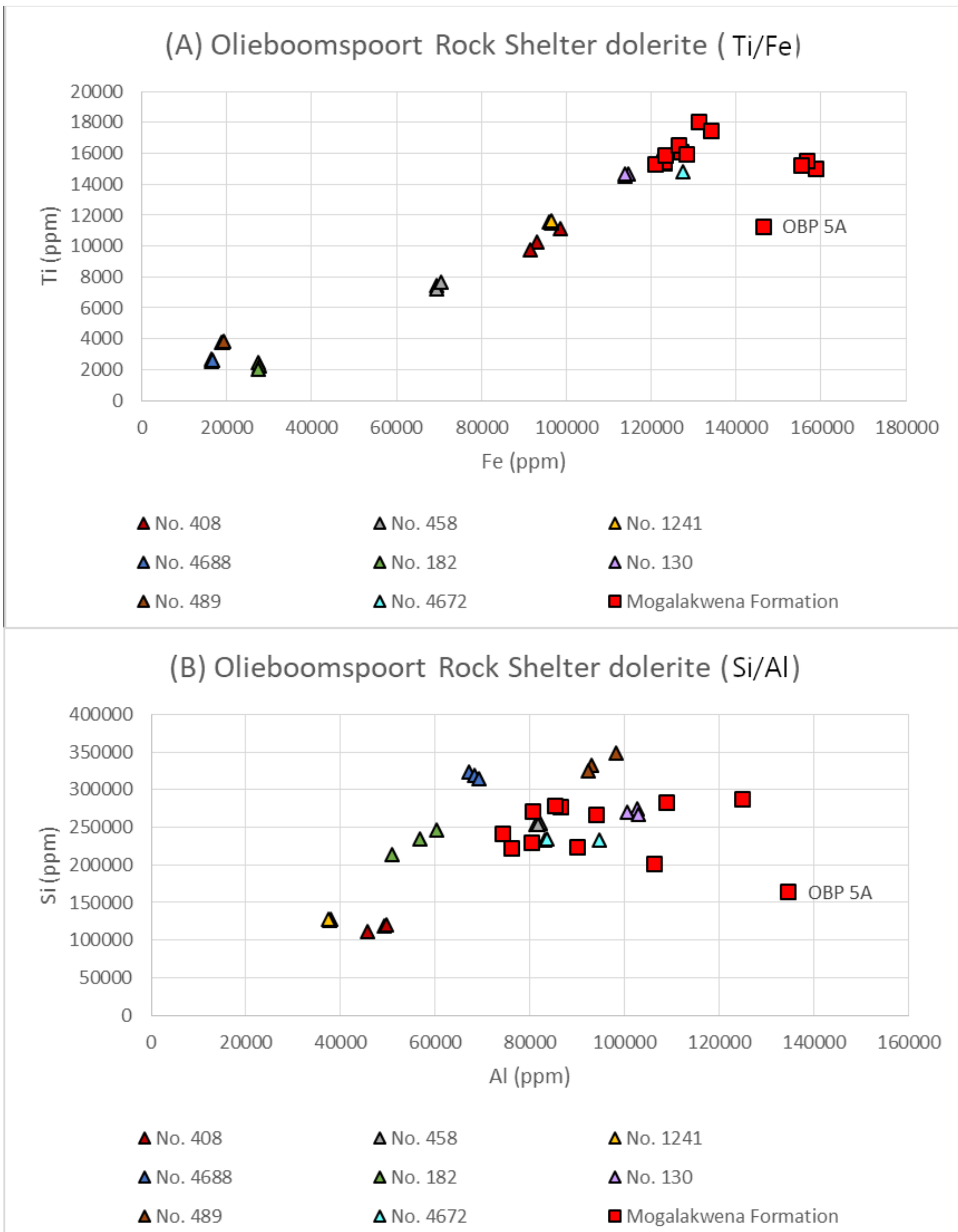


Figure 4.15: A) Ti/Fe scatterplot of dolerite samples at OBP. B) Si/Al scatterplot of dolerite samples at OBP.

Quartz

The Ti/Fe (Figure 4.16 A) and Si/Al (Figure 4.16 B) scatterplots are the most useful graphs for making comparisons between geological samples from the Cleremont Formation and those from the Mogalakwena Formation. Samples from both formations plot very close to one another. The Fe content in the geological samples collected around the Cleremont Formation is less than 4000 ppm and the Ti content is less than 1880 ppm. The Al content of these samples ranges from 1.8% to 3.1% while the Si content ranges from 38.3% to 43.4%. The geological samples collected around the Mogalakwena Formation are more diverse in both scatterplots and generally have higher Fe and Ti contents, as well as Al and Si contents, compared to the Cleremont Formation samples.

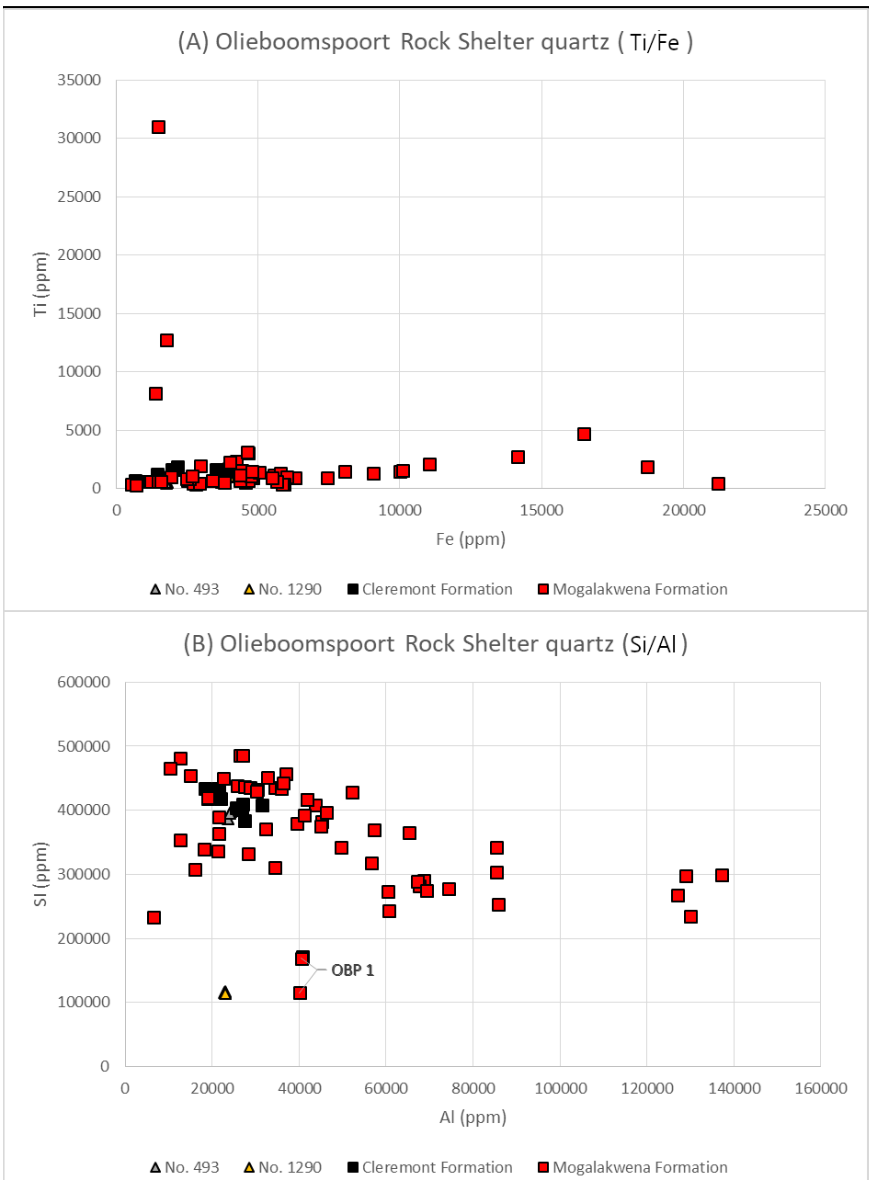


Figure 4.16: A) Ti/Fe scatterplot of quartz samples at OBP. B) Si/Al scatterplot of quartz samples at OBP.

__Principal Component Analysis of Olieboomspoort Rock Shelter Samples__

A total of 48 geological source samples and 65 lithics from around OBP were analysed through pXRF in this project. The results of this analysis were used to differentiate between the different rock types found around the site, and to to see how lithics grouped with the rock types and different foramtions within these rock types. The following elements were used in different PCA plots for this purpose: Al, Si, S, K, Ti, V, Cr, Mn, Cu, Zn, Fe, Ni, RB, Sr, Y, Zr, Nb, Pb, and Th. In all of the following biplots, the first two principle components are plotted. The PCA results are given below and tables with the element coordinates for PC1 and PC2 can be found in Appendix B.

Differentiation of Rock Types

In Figure 4.17, PC1 and PC2 show 61.6% of the total variation obtained through using elements Al, Si, S, Ti, V, Cr, Mn, and Fe. The best projected elements on PC1 (47.5% variation) are Fe, Mn, and V. The best projected elements on PC2 (14.1% variation) are S, Cr, and Si.
Two distinct groups are noted from the Rock Types PCA plot. One with dolerite, and the other with quartzite, quartz, sandstone, and shale. There is less variability in elements best projected by PC2 (S, Cr, and Si) across the different rock types in comparison to those best projected by PC1 (Fe, Mn, and V). Dolerite is richer in Fe, Mn, and V in comparison to quartzite, quartz, sandstone, and shale as seen on the PC1 axis. It can be clearly distinguished from the rest of the OBP rock types. All the rock types, with the exception of dolerite, overlap their 80% significance level ellipses (fill) and their 95% significance ellipses (dash). These rock types cannot be well differentiated from one another based on this plot.

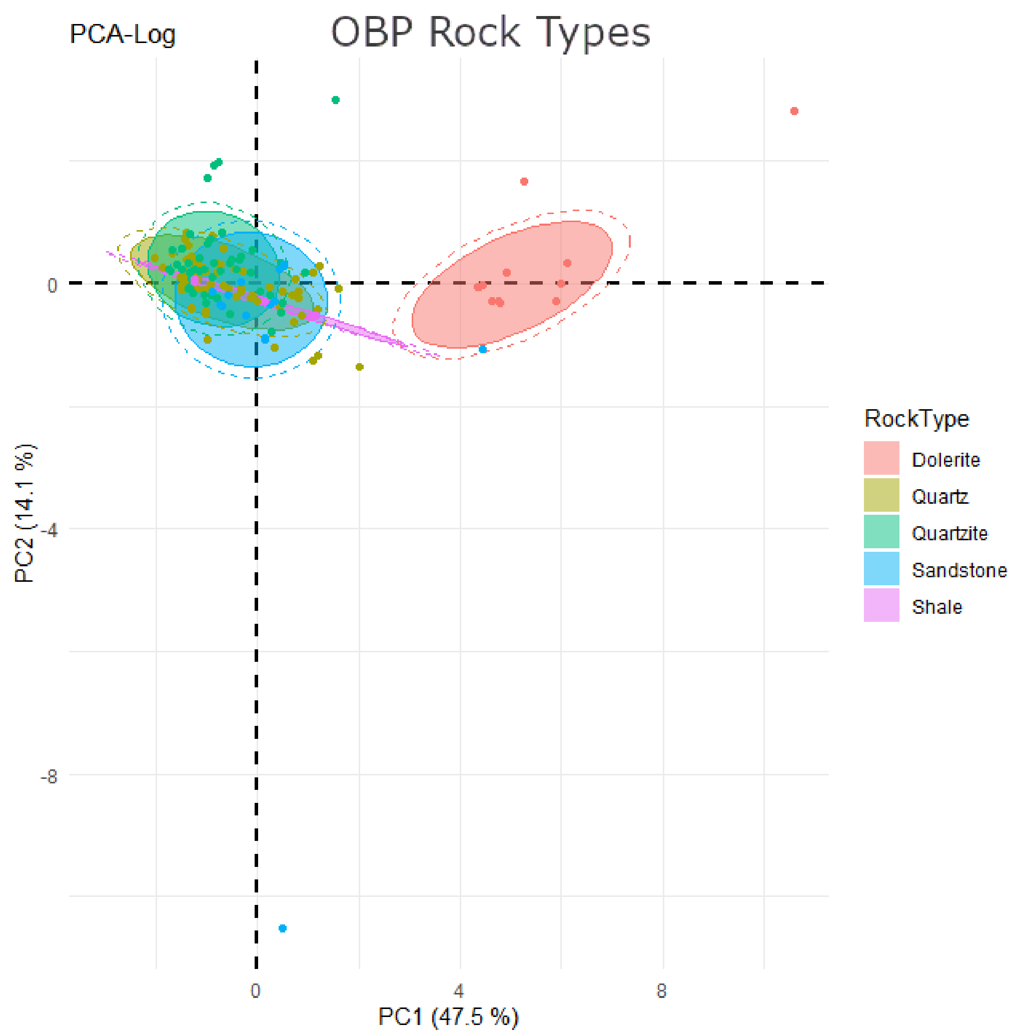


Figure 4.17: The PCA of overlapping OBP rock types (except dolerite) from pXRF data showing 80% significance level ellipses (fill) and 95% significance ellipses (dash).

PCA plots of OBP quartzite samples

In the 'Quartzite Formations' PCA plot, PC1 and PC2 show 53.9% of the total variation (Figure 4.18A). These variations were obtained by using elements Al, Si, Ti, Mn, Fe, Rb, Sr, Y, and Zr. The best projected elements for the quartzite formations on PC1 (35.2% variation) are Si, Fe, Mn and those on PC2 (18.7% variation) are Y, Rb, and Zr.

The Mogalakwena Fm and Cleremont Fm geological samples cannot be differentiated from each other using this plot. Instead the Mogalakwena Fm samples enclose the Cleremont Fm samples with a 100% overlap of its 80% significance level ellipse. The Mogalakwena Fm is more diverse in elements best projected by PC1 and PC2 compared to the Cleremont Fm. Because these formations are overlapping, it is difficult to determine to which the quartzite lithics belong.

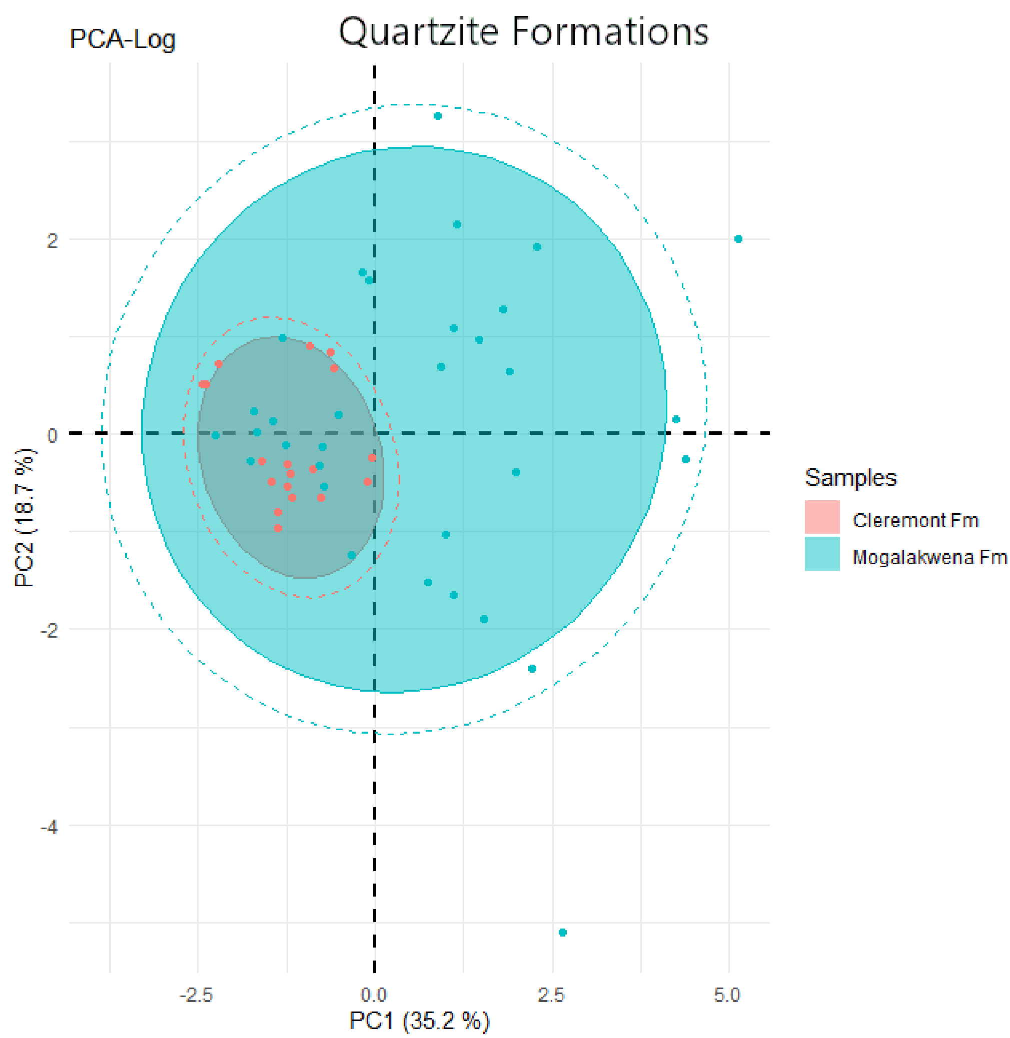


Figure 4.18: The PCA of overlapping OBP quartzite formations from pXRF data showing 80% significance level ellipses (fill) and 95% significance ellipses (dash).

PCA plot of all the OBP shale samples

Only one formation could be studies for shale around OBP. In the ' All shale samples' (outcrops and archaeological samples) PCA plot (Figure 4.19), PC1 and PC2 show 56.4% of the total variation obtained through using elements Al, Si, P, S, K, Ti, V, Mn, Fe, Zn, Rb, Sr, Y, Zr, Nb, and Pb. The best projected elements on PC1 (34.5% variation) are Zr, Nb, and Y. The best projected elements on PC2 (21.9% variation) are S, Zn, and K.

None of the shale lithics, with the exception of No. 420, lie within the the 80% significance level ellipse of the Mogalakwena Fm samples. Instead the lithic assemblage is more diverse along the PC2 axis. This is because insufficient shale geological samples were able to be collected.

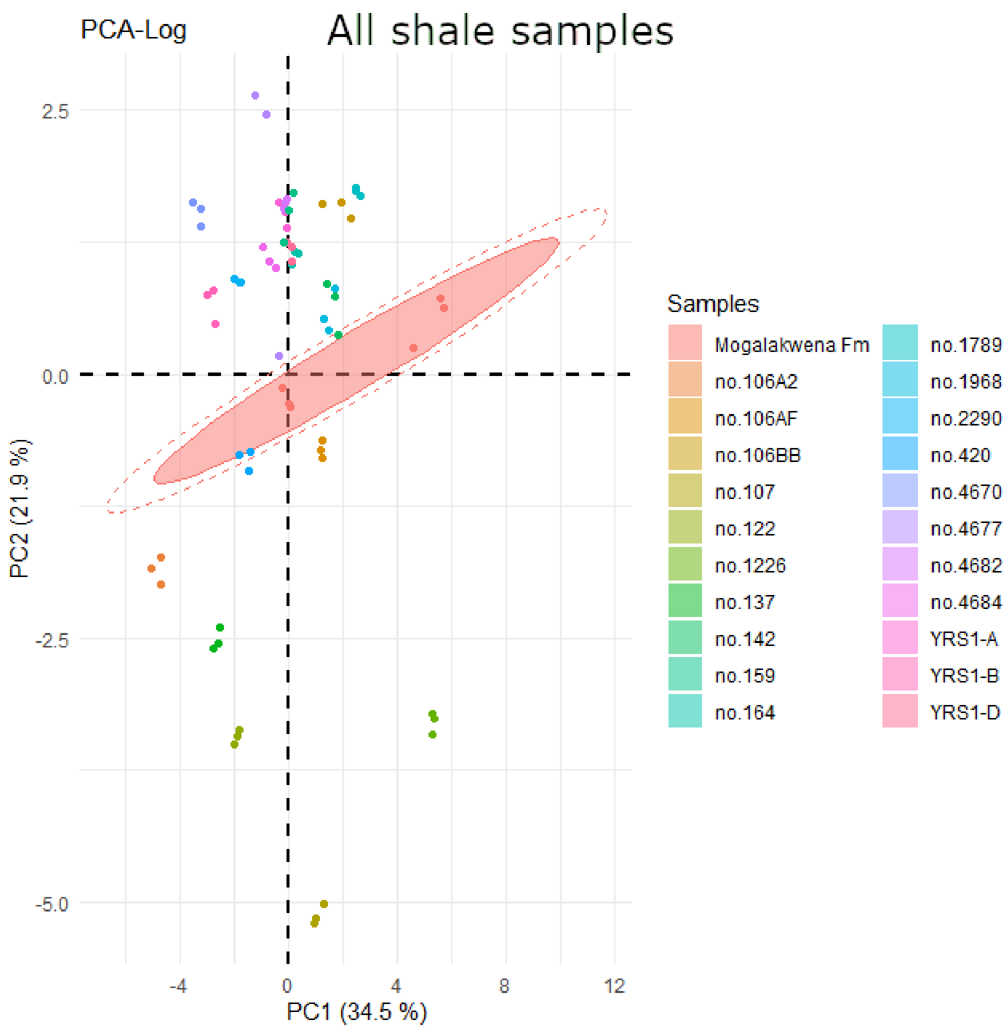


Figure 4.19: The PCA of OBP shale samples from pXRF data showing 80% significance level ellipses (fill) and 95% significance ellipses (dash). It shows the unrelatedness of Mogalakwena Fm samples and the lithics.

PCA plot of sandstone samples

Only sandstones from one formation (Mogalakwena Fm) were analysed in this study. In the 'All sandstone samples' (outcrops and archaeological samples) PCA plot (Figure 4.20), PC1 and PC2 account for 57.3% of the total variation obtained through using elements Al, Si, S, K, Ti, V, Mn, Fe, Cu, Zn, Rb, Sr, Y, and Zr. The best projected elements on PC1 (35.2% variation) are Zn, Zr, and V. The best projected elements on PC2 (22.1% variation) are K, Y, and Rb.

All the OBP sanstone lithics fall within the Mogalakwena Fm 80% significance level ellipse except for No. 1194. No. 1194 is richer in Zn, Zr, and V compared to the rest of the lithics and the geological samples From the Mogalakwena Fm. For this reason, its provenance cannot be determined.

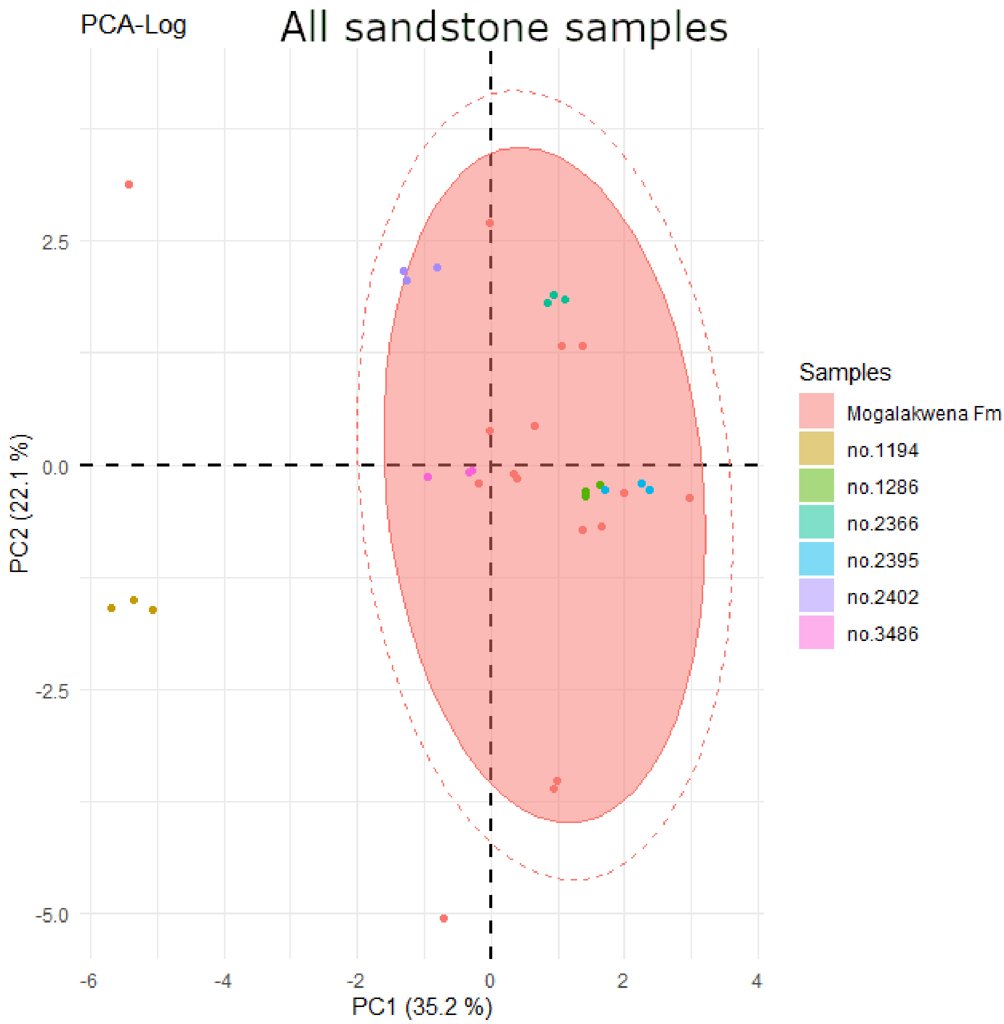
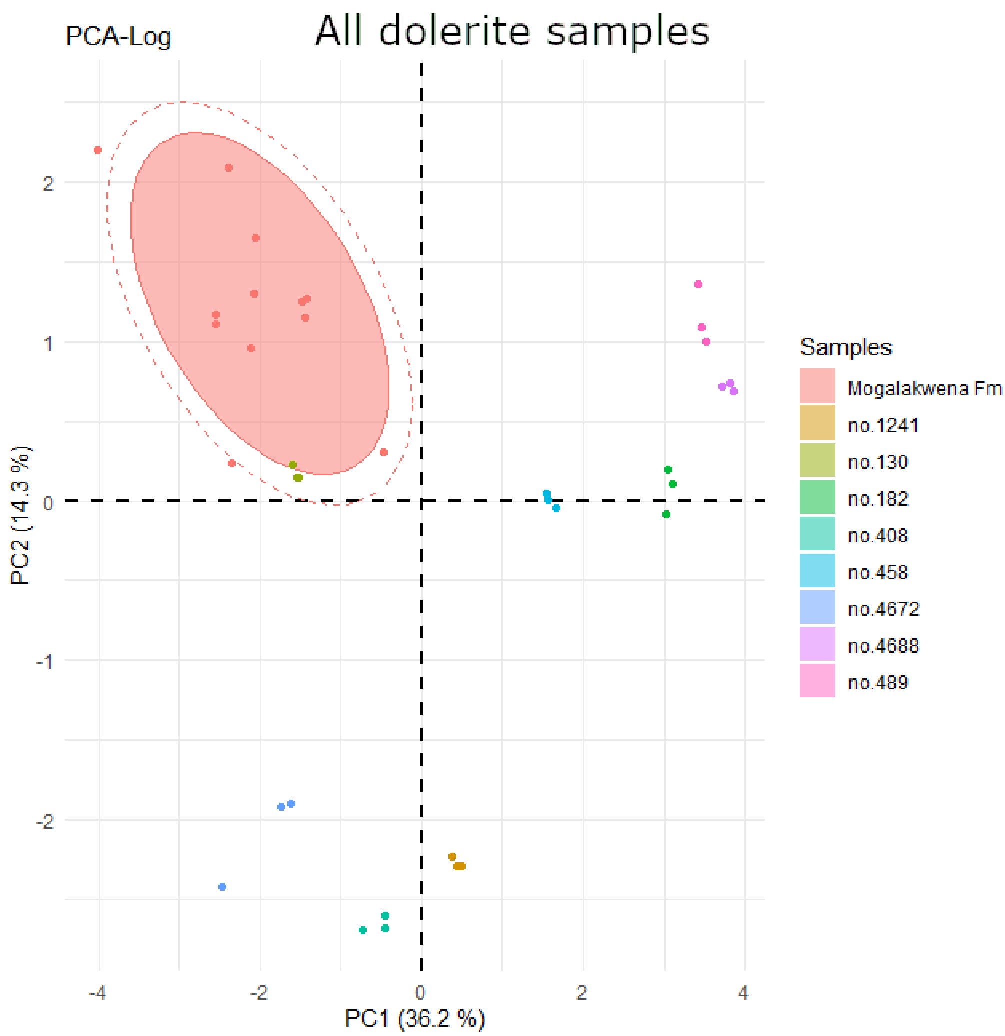


Figure 4.20: The PCA of OBP sandstone samples from pXRF data showing 80% significance level ellipses (fill) and 95% significance ellipses (dash). Most lithics fall within the ellipse of the Mogalakwena Fm samples.

PCA plot of all the OBP dolerite samples

Only dolerites from the Mogalakwena Fm could be analysed in this study. In the ' All dolerite samples' (outcrops and archaeological samples) PCA plot (Figure 4.21), PC1 and PC2 show 50.5% of the total variation obtained through using elements Al, Si, P, S, Ti, V, Mn, Fe, Ni, Zn, Rb, Sr, Y, and Zr. The best projected elements on PC1 (36.2% variation) are Fe, Ti, and Rb. The best projected elements on PC2 (14.3% variation) are P, Si, and Al.

From this PCA plot, it is noted that the majority of the lithics are not similar to the dolerite geological samples collected from the Mogalakwena Fm. Only No. 130 lies slightly on the 80% significance level ellipse of this formation. No. 458, No. 182, No. 4688, and No. 489 are poorer in Fe and Ti, and richer in Rb compared to Mogalakwena shale geological samples. No. 4672, No. 408, and No. 1241 are richer in P and poorer in Si and Al compared to the Mogalakwena shale geological samples. Based on these results it is difficult to determine the provenance of most OBP dolerite



lithics.

Figure 4.21: The PCA of OBP dolerite samples from pXRF data showing 80% significance level ellipses (fill) and 95% significance ellipses (dash). It shows the disparity between Mogalakwena Fm samples and the lithics.

PCA plots of OBP quartz samples

In the 'Quartz Formations' PCA plot, PC1 and PC2 show 58.9% of the total variation (Figue 4.22). These variations were obtained by using elements Al, Si, S, Ti, V, Cr, Mn, and Fe. The best projected elements for quartz formations on PC1 (33.6% variation) are Fe, Mn, and Si and those on PC2 (25.3% variation) are V, Ti, and Al.

The Mogalakwena Fm and Cleremont Fm geological samples cannot be differentiated from each other using this plot. Instead the Mogalakwena Fm samples enclose the Cleremont Fm samples with a 100% overlap of its 80% significance level ellipse, similar to the quartzite geological samples. The Mogalakwena Fm, however, is more diverse in elements best projected by PC1 and PC2 compared to the Cleremont Fm. Due to this overlap, it is difficult to determine the provenance of the quartz lithics from OBP.

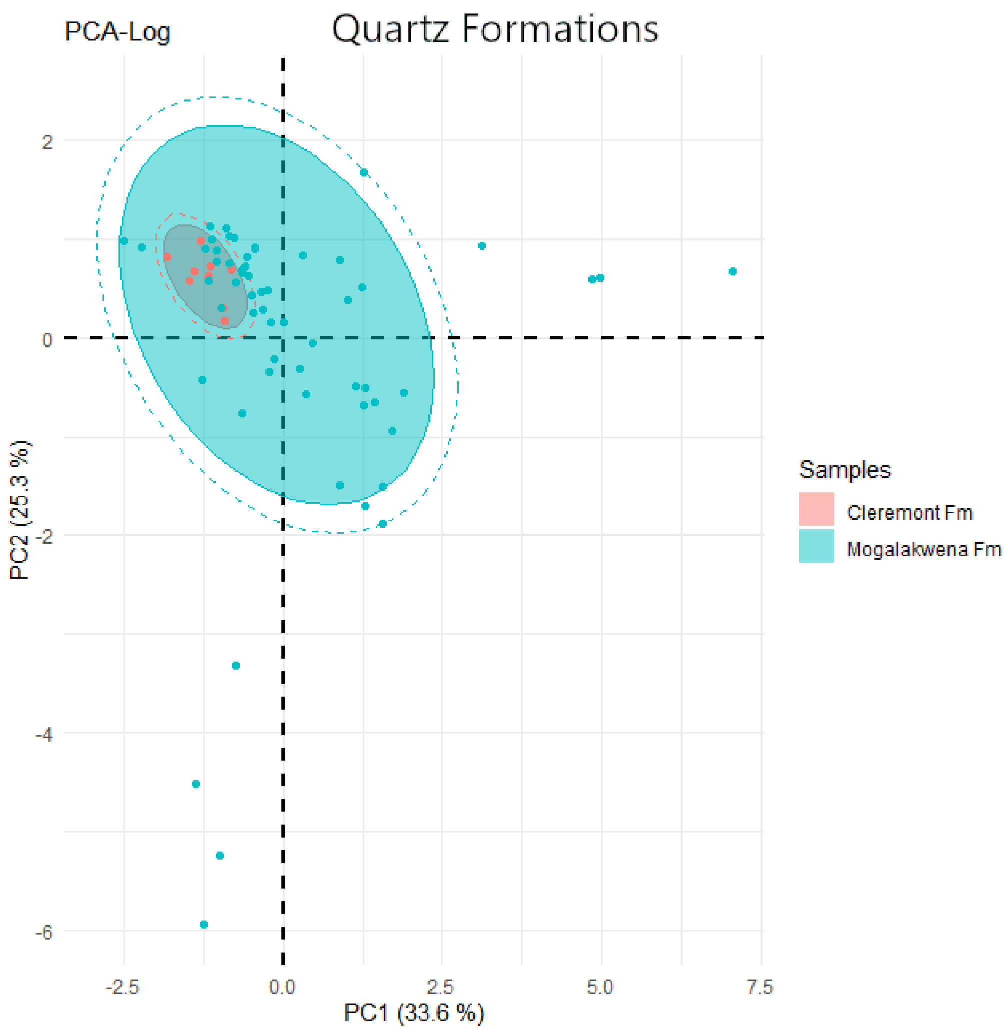


Figure 4.22: The PCA of overlapping OBP quartz formations from pXRF data showing 80% significance level ellipses (fill) and 95% significance ellipses (dash).

Thin sections descriptions

Quartzite (possibly quartz arenite)

No. 486 and No. 3478 (Figure 4.32) are the only two thin sectioned samples that represent quartzite lithics at OBP. They are predominantly composed of quartz grains (~ 90%) in a dark, microcrystalline matrix (<10%). Minor minerals (<5%) such as muscovite are noted in No. 486. The quartz grains in both samples are coarse and have shapes ranging from angular to sub-rounded. Their birefringence is low (grey) and the grains in No. 3478 show both uniform and undulose extinction. The quartz grains in No. 486 only have uniform extinction. The muscovite is colourless under PPL and its cleavage is evident. Under XPL it has high birefringence. Moderate to good sorting is noted in these quartzite samples and they are clast supported. Both samples have interlocking grains with no visible pore spaces. Syntaxial quartz cement in both samples occurs in 40-50% of the quartz grains. The grains in No. 3478 are pitted and fractured and those in No. 486 have deformation lamellae (30-40%). Quartz characteristics used to group quartzite samples are shown in Table 4.1.

Only CMF1 (Figure 4.32 C) has quartzite characteristics. It has around 90% quartz grains in a minimal matrix. They have a low birefringence and 20-30% of them show undulose extinction while the rest show uniform extinction. This quartzite is well-sorted and has interlocking quartz grains. 45% of the grains have quartz syntaxial cement outside of their original boundaries. The grains are pitted and about 25% of them have embayments. Black staining is noted throughout the sample and very little deformation lamellae are present. Quartz characteristics used to group quartzite samples are shown in Table 4.1.

Table 4.1: Quartz characteristics of quartzite samples at Olieboomspoort Rock Shelter. **OBP Quartz Characteristics**

Sample no.	**Undulose extinction**	**Deformation lamellae**	**Concave-convex boundary relationships**	**Syntaxial cement**	**Stylolites**	**Interloc-king quartz**
No. 486	25%	30-40%	85%	40%	-	90%
No. 3478	20%	-	70%	40-50%	-	>90%
CMF1	20-30%	<10%	75%	45%	-	>90%

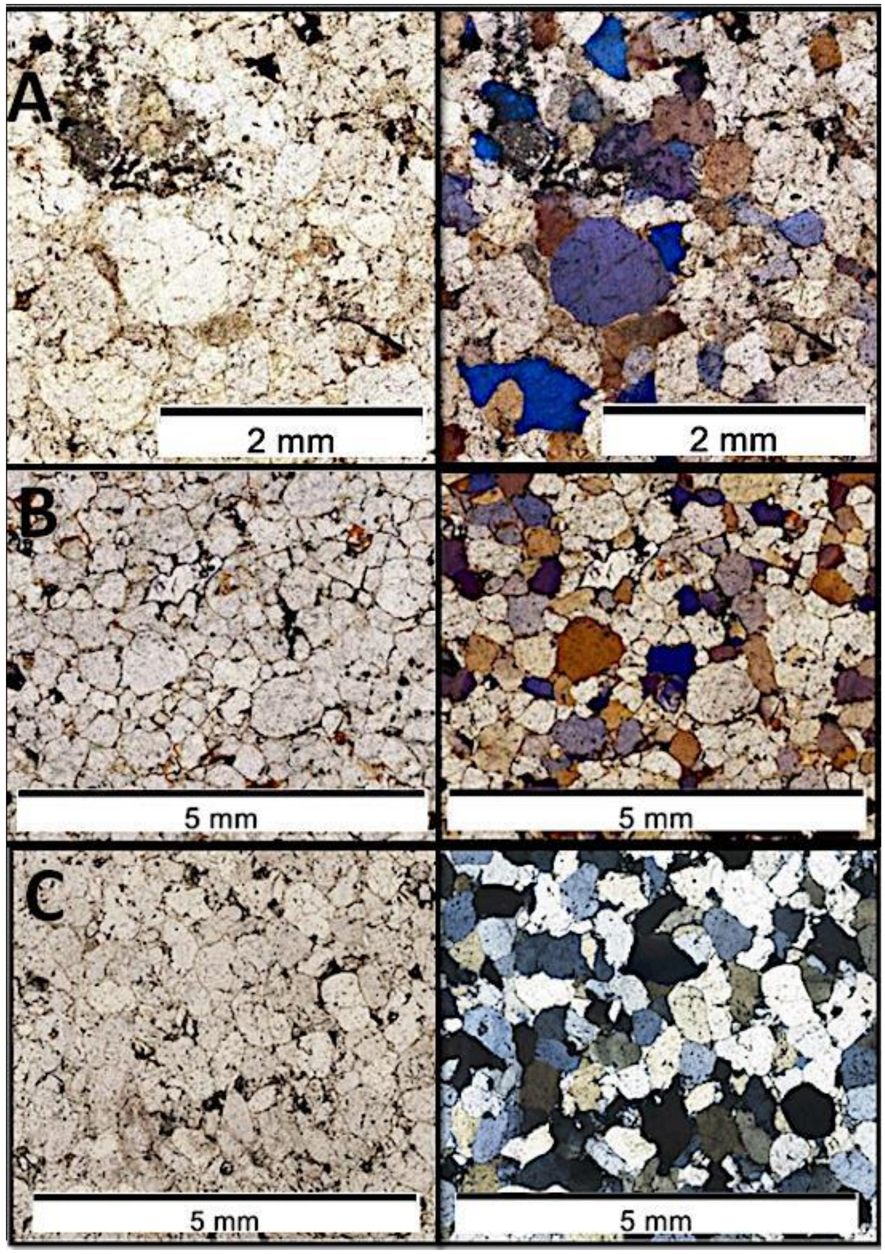


Figure 4.32: Light microscope image of A) No. 486 showing interlocking quartz grains, B) No. 3478 showing sub-rounded and interlocking quartz grains, and C) CMF1 showing its interlocking and annealing quartz grains in PPL (left) and XPL (right).

Sandstone

No. 1194 is a greywacke and No. 2402 is a sub-litharenite (Figure 4.33). Both samples have 60-80% quartz grains in a visible matrix (20% in 1194). Other minerals like muscovite and lithic fragments are noted in No. 2402, and opaque minerals are present in No. 1194. No. 1194 has quartz phenoclasts. The quartz grains in this rock type are angular, sub-angular, and sub-rounded. They have low grey interference colours and show both undulose and uniform extinction. Both samples have recrystallized quartz grains (10-40%). The muscovite in No. 2402 has visible cleavages and bright interference colours under XPL. The lithic fragments are multi-coloured under XPL. These sandstones are poorly sorted. The quartz grains are pitted and fractured, and those in no. 2402 have deformation lamellae (20%).

Two sandstones are identified in the geology around OBP. These are arkose (OBP 7; Figure 4.33 C) and greywacke (OBP 8A; Figure 4.33 D). A similar amount of quartz is noted in both sandstones (55-60%). Alkali feldspar (40%) is a component of the arkose, whereas muscovite (20%) and opaque minerals (5%) in a significant matrix (20%) form part of the greywacke mineralogy. Both samples have a degree of irregularly shaped quartz grains amidst the sub-rounded, or angular to sub-angular grains. All of these have a low birefringence and uniform extinction although OBP 8A shows undulose extinction too (55%). 45% of the quartz grains in OBP 7 have been recrystallised. The alkali feldspar in OBP 7 is a cloudy brown under PPL and has a low birefringence. The muscovite in OBP 7 has a high birefringence and becomes extinct parallel to its perfect cleavage. Both samples are poorly sorted. In OBP 7, the alkali feldspar has a perthitic texture. Black and orange staining, is noted in association with the grains throughout the sample (OBP 7). The grains in OBP 7 are pitted (90%) and fractured (40-50%). In OBP 8A, 25% of the quartz grains have deformation lamellae.

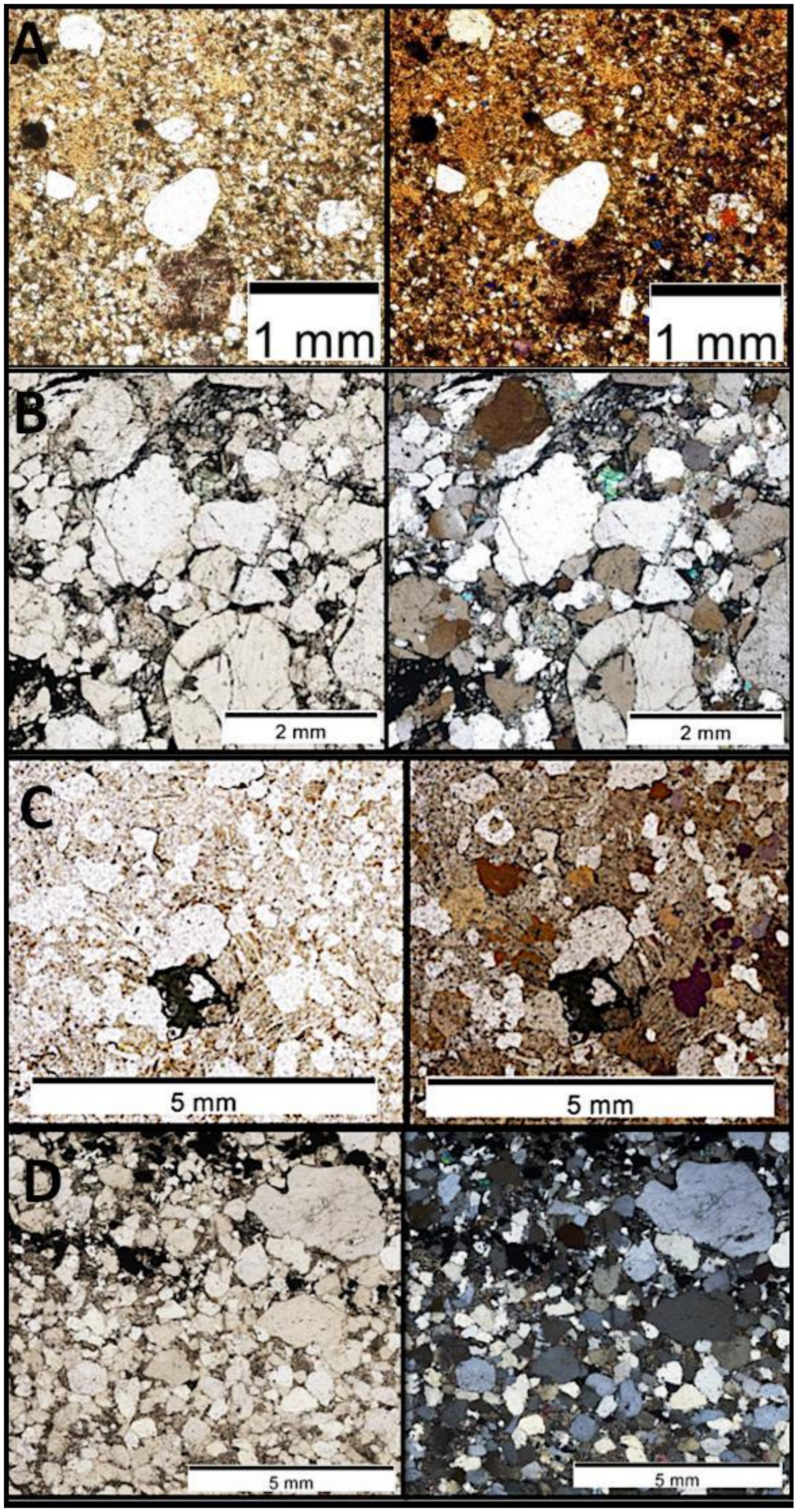


Figure 4.33: Light microscope image of A) No. 1194 showing its poor sorting and quartz phenoclasts, and B) No. 2402 showing the poorly sorted nature of the sandstone in PPL (left) and XPL (right). C) OBP 7 shows the relationship between quartz grains and alkali feldspar grains in PPL and D) is OBP 8A showing its porosity and sorting of grains in PPL (left) and XPL (right).

Shale

No. 164 and YRS1-D (Figure 4.34) are very fine-grained shale samples. The only minerals large enough to analyse in both these samples are opaque minerals (10-15%) and chlorite (20-25%). The chlorite has a low birefringence (brown anomalous interference colour) under XPL. Chlorite occurs in clusters in both samples. In No. 164, it surrounds the opaque minerals throughout the sample.

OBP 10A (Figure 4.34 C) is composed of 20% opaque minerals in a very fine-grained, green-brown matrix (80%). It has cloudy patches of opaque mineralisations in some parts of the sample.

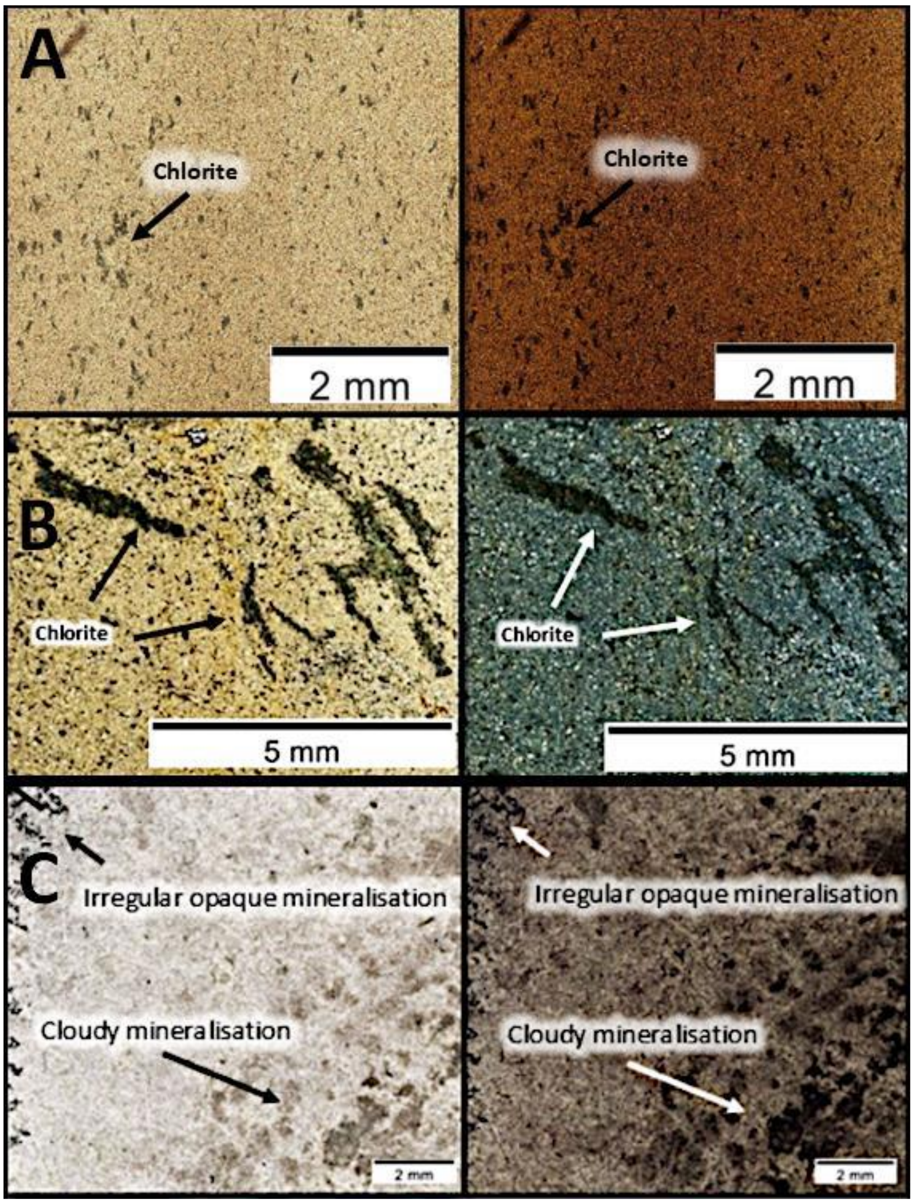


Figure 4.34: Light microscope image of A) No. 164, B) YRS1-D showing cryptocrystalline texture and chlorite, and C) OBP 10A showing its cryptocrystalline texture, and cloudy, opaque mineralisation in PPL (left) and XPL (right).

Dolerite

The only thin sectioned dolerite sample is No. 458 (Figure 4.35 A). It has white plagioclase laths (80%) in a black matrix. Other finer minerals like hornblende and augite are also noted in this sample through their bright blue, yellow, and green interference colours under XPL. The plagioclase laths have a low birefringence and are randomly oriented throughout the sample.
OBP 4G (Figure 4.35 B) has mineralogy consisting of plagioclase laths (80%), white euhedral phenocrysts (5%), and brown biotite (15%). The plagioclase laths and phenocrysts have a low birefringence whereas the biotite shows third-order orange-yellow interference colours under XPL. These minerals are randomly orientated throughout the sample. The phenocrysts are altered and have brown mineralisation along their grain boundaries.

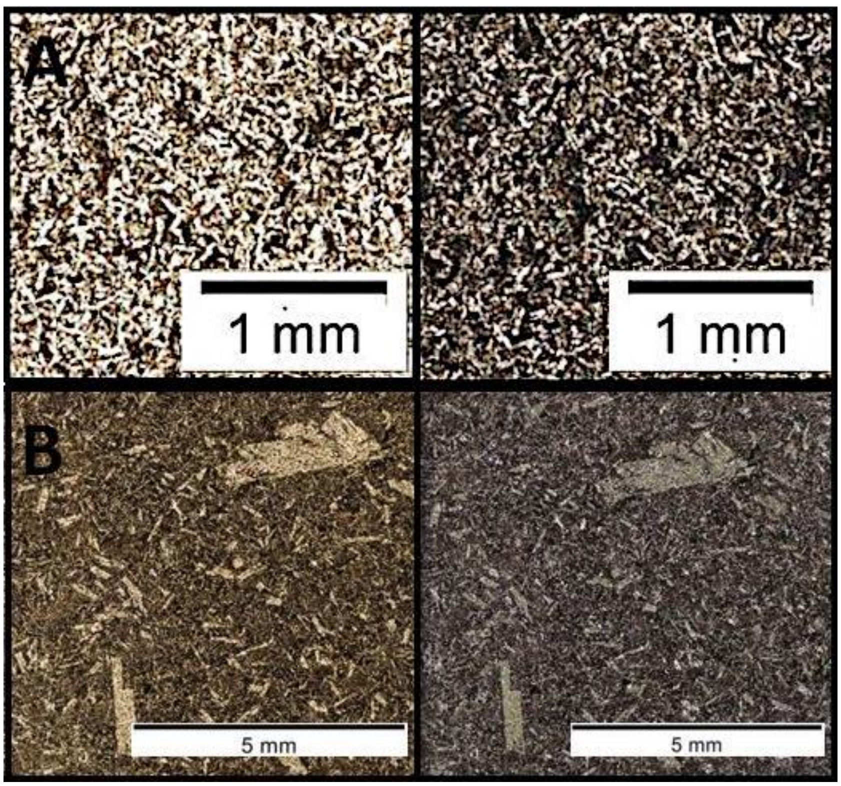


Figure 4.35: Light microscope image of A) No. 458 and B) OBP 4G showing randomly oriented plagioclase laths in PPL (left) and PXPL (right).