Semi-quantitative petrographic characterization of Bronze Age quartzite tools – a multivariate approach

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Text of abstract

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Highlights: These are the highlights.

# 1 Introduction

The last decades have seen an increase in the interest for non-flint lithics and raw material studies in European archaeology, providing much needed development in a previously neglected field (Blomme et al. 2012; Dalpra and Pitblado 2016; Prieto et al. 2019; Ramacciotti et al. 2019; Prieto et al. 2020, 2021). In regions where local flint and obsidian sources are non-existent, such as Northern Fennoscandia, other materials have been the primary target for lithic production[[1]](#footnote-21) (e.g. quartz, quartzite, slate) (Baudou 1978; Rankama et al. 2006; Olausson et al. 2012). With the lack of any natural geological source of flint in Northern Sweden both quartz and quartzite are instrumental for the inference of settlement and mobility within the region. While quartz and quartzite technology have been part of a number of studies over the years, there have been less development in the petrographic characterization and provenience studies of these same materials (Broadbent 1979; Callahan et al. 1992; Tallavaara et al. 2010; Knutsson et al. 2016). One reason for the lack of methodological development in North Swedish research could be the complex geological background of the region and the abundance of moraine formations (Bargel 2003; Stroeven et al. 2016; Stephens and Bergman Weihed 2020). The degree to which geological materials have been mixed and transported demands considerable effort in order to reconstruct the events that have formed the modern day landscape. While tentaive interpretations of lithic material sourcing tends to describe strategies leaning towards direct procurement from the mountain zone (Forsberg 1985, 2012), both quartz and quartzite could potentially have been sourced locally from exposed veins in the bedrock, or as loose rocks and pebbles in the numerous moraine formations, as part of a strategy leaning more towards an embedded adaptation (Binford 1979; Rankama et al. 2006, pp. 249–250). Due to this ambiguous nature of availability of raw materials provenience studies are complex and fraught with uncertainty.

Prieto et al. (2019, p. 15; 2020, p. 32) notes that the usage of the term *quartzite* in archaeology has been somewhat ambiguous, often used interchangeably between sedimentary orthoquartzite and metamorphic quartzite. It is difficult to provide a comprehensive definition of quartzite, owing much to its metamorphic nature, and different researchers have thus emphasized different features as being characteristic of the material (Howard 2005). Visual characterization on a megascopic level is typically unreliable, and microscopic analysis is recommended for the identification of metamorphic structures, but Howard (2005, p. 708) also emphasises the need for field-based methodologies. In the case of archaeological research it is worth considering situations where destructive techniques simply are not applicable. Artefact collections (museum, private, etc.) represent a valuable resource and asset, with easy access to large datasets with a wide geographical distribution. While destructive methods may prohibit access to such material there are still non-destructive alternatives that can generate valuable information. Aside from being non-destructive spectroscopic instrumentation feature fast measurements, making it an attractive option for field-based screening purposes. While X-Ray Fluorescence (XRF) is the go-to spectroscopic technique for petrographic characterization (Tykot 2003; Shackley 2011a), due to its capability of providing compositional data, there are other instrumentations that may provide a deeper insight into the structural properties. The use of near-infrared spectroscopy (NIRS) goes almost as far back as the XRF (Hunt 1977), and there is an impressive body of work dedicated to the fingerprinting of geological materials (Linderholm and Geladi 2014a, b; Sciuto 2019). The same can be said for Raman spectroscopy, which has been applied extensively in the Cultural Heritage sector and in the analysis of artwork and manuscripts (Vandenabeele 2012).

## 1.1 Aims

In the current study a dataset of quartz and quartzite artefacts from museum collections in Västerbotten, Sweden, have been analysed using the three above mentioned spectroscopic instrumentations (NIRS, Raman and XRF). The aim of the study is to evaluate their viability for fingerprinting quartz and quartzite material in the North Swedish region based on surface measurements. The study is a part of a larger project wherein models of settlement and mobility are being evaluated and developed in regards to raw material procurement and use. As flint is a non-local material to Northern Sweden it follows that any find of this material within the region most likely have been brought there through anthropogenic activity. In contrast, determining the degree to which cultural and/or environmental factors have influenced the distribution pattern of quartz and quartzite artefacts, however, is far more difficult. While there is a good understanding of the geological makeup of the Scandes (Stephens and Bergman Weihed 2020), the quartzite itself remains largely uncharacterized, with few reference samples available for spectral comparison. The glacial impact on the landscape further compounds the issue as geological material has been transported and deposited by glacial movements, as well as subsequent postglacial processes. Although models for settlement and mobility can be used to test ideas of material distribution as influenced by cultural processes (e.g. Forsberg 1985), it is also necessary to attempt to estimate the degree of environmental impact on this pattern.

In order to engage in any meaningful analysis, however, it is first necessary to distinguish between different quartz and quartzite groups. Principal Component Analysis (PCA) has been used to model the spectral data in order to discern material groupings related to different spectral features and their contributions to the Principal Components (PC). The results form the basis of a discussion of the viability of using non-destructive spectroscopy in petrographic characterization of quartz and quartzite material, and thus future efforts of developing models for raw material sourcing in Northern Sweden.

# 2 Material

The current study is based on spectroscopic surface measurements collected on quartz and quartzite points recovered from sites within the County of Västerbotten, Sweden. A dataset of 444 points, point fragments and preforms made from quartz and quartzite material stored at the collections at Västerbotten (www.vbm.se) and Skellefteå (www.skellefteamuseum.se) museums were analysed using NIRS, raman and XRF spectroscopy. The material features a mainly black, grey or white hue, as well as a subset of semi-translucent material with a beige tint to almost fully translucent. The colour of the material has been classified both according to the Munsell system, as well as sorted into general categories of hue for visualization purposes: *dark* (~ N1-N4), *light* (~ N5-N8), *white* (~ N9), *colourless* (lack of colour, opaque to translucent). A number of these points have, according to prior classifications, been worked using bifacial technology. While there currently is no local chronology available for this artefact type in the North Swedish region[[2]](#footnote-24), there have been attempts (Forsberg 1985, p. 5, 1989; Baudou 1992, p. 99). Previous research suggests a period of use that falls between ca. 3 800 - 2 000 BP (Käck 2009, p. 60). Similarly, studies of raw material use at sites dating to this period seems to indicate an increase in the use of quartzite (Baudou 1978).

A large portion of the collections stored at North Swedish museums were recovered from excavations and surveys performed in conjunction with the river regulations during the 1940s – 1980s (Janson and Hvarfner 1960; Biörnstad 2006), as well as subsequent follow-up survey projects (e.g. Andersson 2015). This has likely had a contributing factor to the distribution pattern of the artefacts observed in fig. 2.1 as the known sites with finds of points and preforms are concentrated around the parishes of Dorotea, Lycksele[[3]](#footnote-25), Vilhelmina and Tärnaby. This can be seen more clearly in fig. 2.2 with the four parishes accounting for almost ¾ of the total no. of samples.

Västerbotten museum have come far in their digitization effort, making large portions of their collections searchable online. Based on a query of all points and preforms labelled as *quartz*, *brecciated quartz* or *quartzite* a list was generated from their database. Sampling of this list of artefacts was not random. Sites located in areas with few finds were included first in order to increase the geographical distribution, with sampling of areas with higher site density making up the remaining bulk of the dataset. None of these were from the northernmost Skellefte river, however, and thus a selection of 5 artefacts each from three well-known sites stored at the collections of Skellefte museum were included. This resulted in a total dataset of 497 artefacts, and after documentation and looking up missing information, 444 of these were determined to be either points or preforms made from quartz or quartzite material.

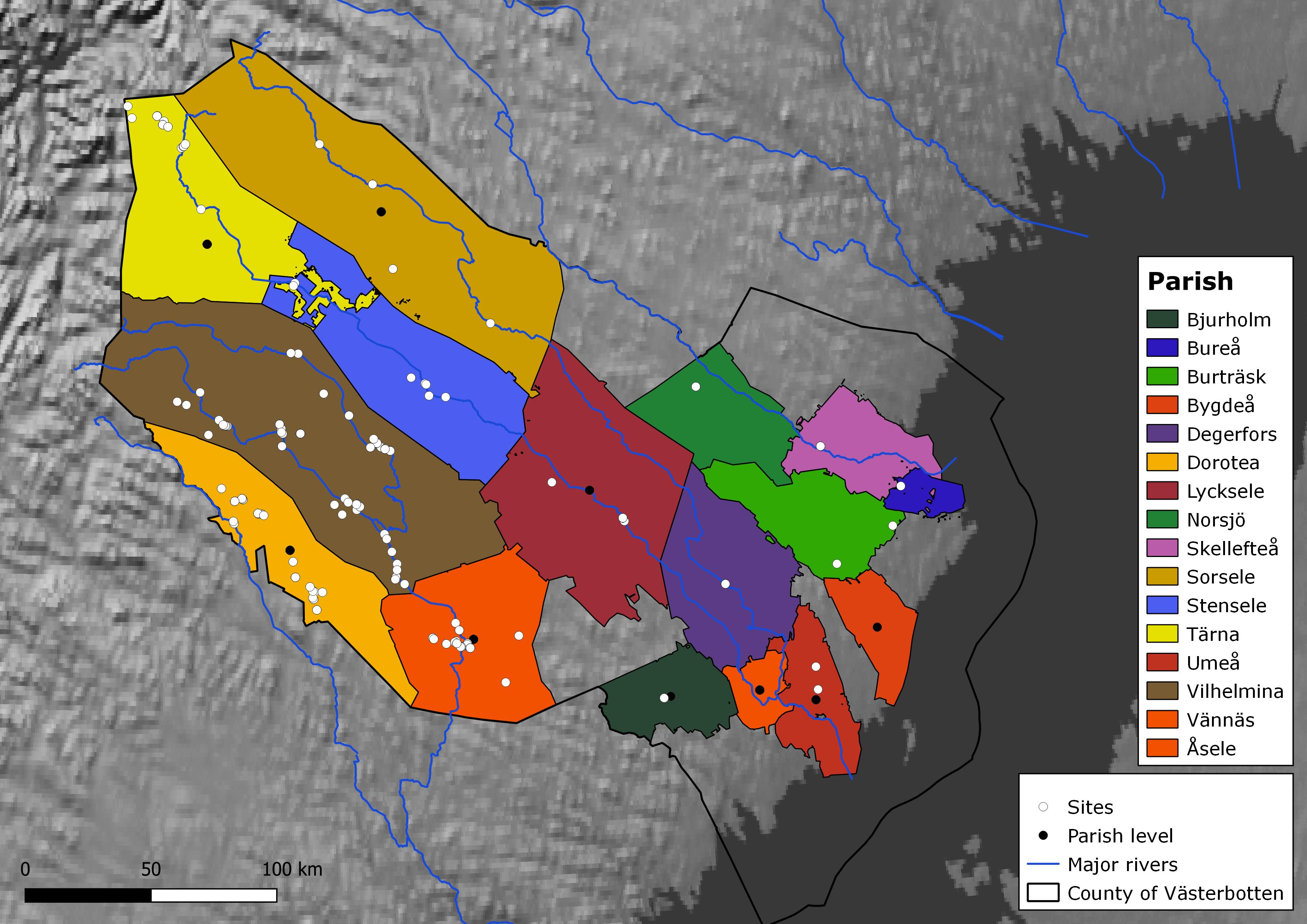


Figure 2.1: Geographical distribution of sampled sites within Västerbotten County, Sweden. The distribution of sampled sites is likely affected by the excavation and survey history of the region. White: Sampled sites; Black: Parish centroid for measured samples lacking site coordinates. Made with Lantmäteriet (CC0), Natural Earth, and Riksantikvarieämbetet.

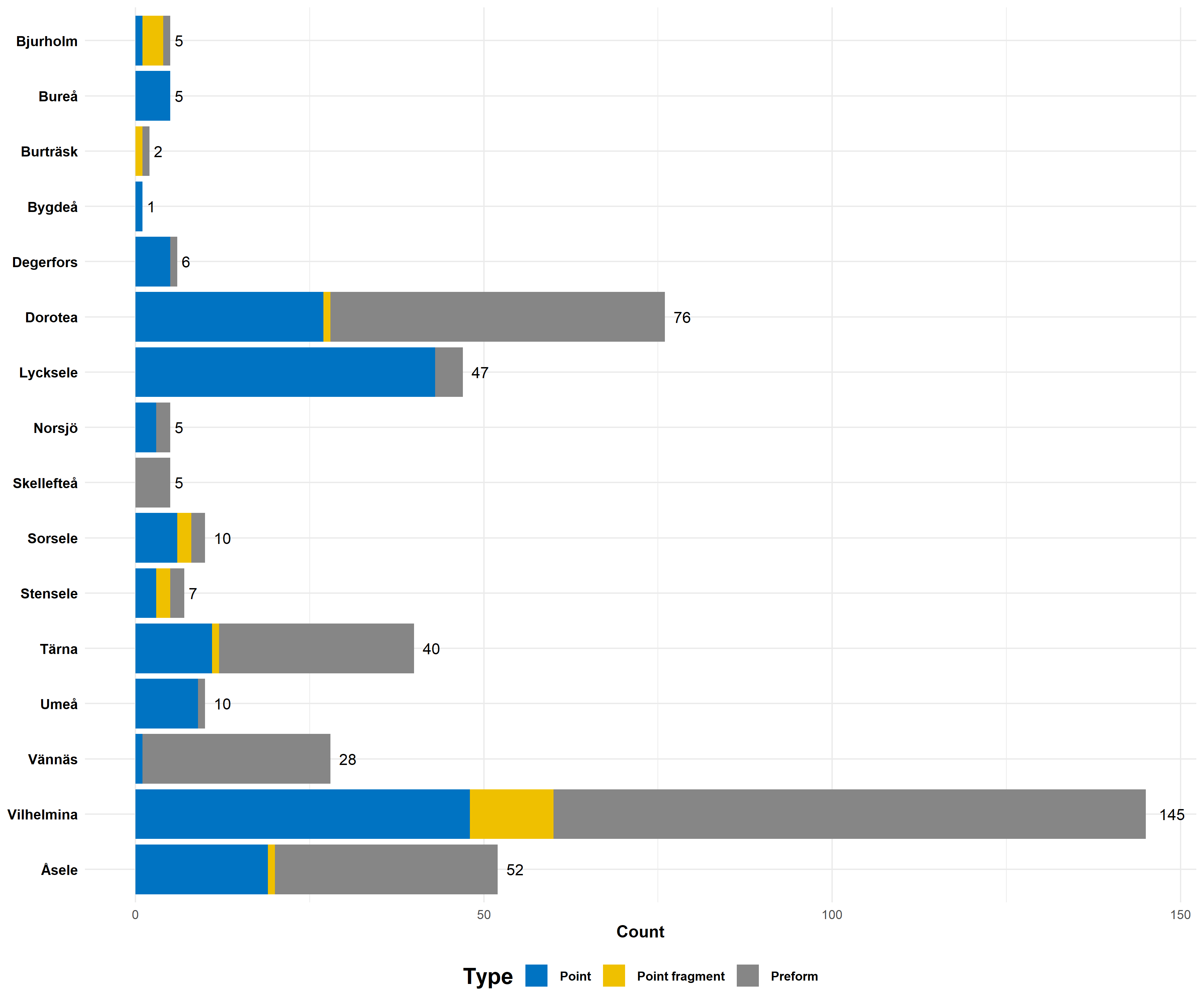


Figure 2.2: Distribution of the 444 points and preforms across the parishes in Västerbotten, Sweden. Note that while Lycksele feature a large number of samples 41 of these are from the site of Lycksele 301

While parts of the collections at Västerbotten museum have been petrographically analysed by a geologist (Åhman 1967; Biörnstad 1968), at this point it is difficult to say with any certainty how many (if any) of the artefacts in the current dataset were among these. It is fair to assume that the vast majority have been classified visually based on megascopic features according to a “standard” archaeological nomenclature. In the dataset the four main types of registered materials are: quartz, quartzite, brecciated quartz and brecciated quartzite[[4]](#footnote-28). Those that had no prior material classification registered were sorted into either the quartz or quartzite category. The reason for including quartz in the study is in order to test whether it is possible to separate quartz from quartzite using these methods, as well as a way to account for potential mistakes in the original classification.

Another reason for keeping a quartz category is the group referred to as *brecciated quartz*. In a petrographic study of the artefacts recovered along the Ume river valley Åhman (1967, p. 8) briefly touches upon the definition of *brecciated quartz*, also referring to it as a *mylonite quartz*. Åhman describes the material as having formed from a quartz vein which has fractured due to movements in the mountain massives and recrystallized via pressure. While the translucent material may give the impression of an unusually pure quartzite, Åhman states that the thin-section analysis reveals the fragmented configuration of quartz. *Brecciated quartz* is a somewhat common term used in North Swedish archaeology, and has typically been used to identify a certain translucent type of fine-grained, quartz-rich material (e.g. Holm 1991, p. 24).

# 3 Methods

All measurements were performed at the Environmental Archaeology Lab at Umeå University[[5]](#footnote-30). The analysis was conducted in a dark room in order to minimize light pollution, with the main light sources in the room being the computer screens. Each artefact was measured twice, on different sides, with additional measurements in cases where sampling was difficult. The only exception to this was the XRF, where due to time constraints one measurement was collected as a rule apart from a few exceptions. This lowers the statistical reliability of the XRF analysis and inferences of the results should be evaluated with this in mind. The size of the dataset alleviates some of these issues, as for the larger sites there are multiple objects that may be of the same material, however, for the smaller sites there still remains some uncertainties. For materials that featured high heterogeneity the sample point was focused, as much as possible, on the surrounding matrix.

Pre-processing of the spectra is different for each instrumentation and based on relevant literature. After pre-processing the data was modelled using PCA, which is an unsupervised exploratory technique for reducing the dimensionality of the dataset (Geladi and Linderholm 2020). PCA modelling was performed using the base R *prcomp()* function (R Core Team 2017, 2021).

## 3.1 Near Infrared

NIRS measures the absorbance of light within the near infrared region (780 nm – 2 500 nm) of the electromagnetic spectrum. Based on the theory of molecular vibration and excitation (Hunt 1977), by exposing a geological material to electromagnetic radiation it is possible to generate overtone vibrations which can be related to certain molecular groups. The most common group capable of generating these overtones is the OH group, which in quartz-based materials typically relates to molecular water (Hunt 1977, p. 508).

NIR analysis was conducted with the Analytical Spectral Device (ASD) LabSpec 4, using a contact probe. The ASD features a detection range of 350 nm – 2 500 nm, with spectral sampling (resp. spectral resolution) of 1.4 nm (resp. 3 nm) in the visible and near infrared range and 1.1 nm (resp. 10 nm) in the short‐wave infrared range. Each object was measured against a white reference background and the collected measurements were averaged for each artefact. The visible spectrum (~ 400 nm - 750 nm) and the NIR range (~ 1 000 nm – 2 500 nm) were analysed separately, with the former focused on colour analysis and the latter the chemical structure of the material. The data was mean-centred before PCA modelling.

## 3.2 Raman

Raman spectroscopy shares some similarities with NIRS in that they both can be used to infer molecular structure based on their vibrational properties (Nafie 2001). However, where NIRS measures light absorbance and generate overtones of molecular vibration, Raman spectroscopy is based on the *Raman scattering* effect. By exposing a molecule to radiation with a monochromatic laser it is possible to induce a dipole moment in the molecule which generates the Raman effect. The subsequent light that is scattered contains both Rayleigh scatter and Raman scatter, the former at a frequency of incident radiation and the latter at a shifted frequency (Dubessy 2012). The spectrometer will measure the Raman scatter and generate a spectrum where the spectral line has shifted compared to the incident Rayleigh frequency. As the structure of a molecule will inform its vibrational mode, the point at which these shifts occur within the infrared region varies depending on the material (Smith and Carabatos-Nédelec 2001).

The Raman instrumentation used was a portable i-Raman EX featuring a 1064 nm excitation laser. The spectral coverage of the instrumentation is 175 cm-1 – 2 500 cm-1[[6]](#footnote-32) with a resolution of 9.5 cm-1. Access to the spectrometer was made possible by the department of Forest Biomaterials and Technology at the Swedish University of Agricultural Sciences (SLU) in Umeå[[7]](#footnote-33).

After averaging the spectra for each artefact the results were baseline corrected in order to detrend the data. This was achieved using modified polynomial fitting in R (R Core Team 2017) with the ChemoSpec package (Hanson 2016). The spectra were then mean-centred and normalized using Standard Normal Variate (Shaver 2001, pp. 284–285) before PCA.

## 3.3 X-Ray Fluourescence

XRF spectroscopy, and its capabilities for quantifying chemical elements in different materials, has made it a well-established method for petrographic analysis in archaeology (Hall 1960; Shackley 2011a). Exposing a material to X-ray radiation causes electrons to dislodge and move between the electron shells of the atoms, which in turn emits energy (i.e. *fluourescence*). As the energy differences between electron shells for the various elements is known it is possible to both identify and quantify the abundance of an element (Shackley 2011b, p. 16), which is useful for provenience studies where the chemical character of an unknown and a reference sample can tested statistically (Tykot 2003).

XRF measurements were collected using a Thermo Scientific Niton XL5 Analyzer, connected to a portable test stand. The reference calibration used for element quantification was set to *mining* mode. Sampling with this setting takes 2 minutes in order for the instrument to cycle through all of the filters (main, low, high, light). Due to time constraints and the number of objects in the current dataset it was only possible to collect one measurement on each object, apart from some exceptions.

The XRF data was log-transformed, mean-centered and scaled to unit variance before PCA modelling. As the resulting dataset also features values below the Limit of Detection (LOD), these were treated as NULL (or “missing values”), during PCA. Since missing values are not permitted during PCA these had to be imputed before the analysis, which was performed using the *missMDA* R package (Josse and Husson 2016) developed for datasets with incomplete data. Two methods for imputation can be implemented, iterative or regularized, and since the former suffers when there is a large number of missing values the latter was used.

# 4 Results

## 4.1 NIR - visual range

Examining the average spectra of the different colour groups (dark, light, white and colourless), as expected, the darker material feature higher absorbance of light than the other groups. No other distinctive features can be seen in the visual band, that could potentially be used to distinguish them. It is, however, apparent that while the more extreme hues of dark and white material can be isolated more clearly based on their spectra, there also exists a wider greyscale of material that does not conform as easily to these broader groups.

The cumulative explained variance for the first three components in the PCA is ~ 99.9%, with the accompanying loadings in fig. 4.1. Most of the contribution to the first Principal Component (PC) can be found within the red part of the spectrum (~ 600 nm - 750 nm). This can be seen in the score plot where darker and lighter materials are separated along PC 1, as darker samples would feature a higher absorbance within the red part of the spectrum (fig. 4.2). In contrast, most of the contribution to PC 2 occurs within the blue-violet part of the spectrum, which does not seem to alter the structure of the dataset in any significant way. PC 3 feature high contributions within both the blue-violet and red parts of the spectrum, which also seems to draw the light and dark samples that feature a blue-gray tint towards the top of the score plot.

While the overall structure of the model seems to group colourless and white materials towards the bottom left, with dark material to the right, there are a number of samples that break this pattern. This suggests that the perceived colour of a material does not necessarily match what is detected within the visible spectrum of light. It is also difficult to separate out the greyscale of colours that occur inbetween the “black and white” groups, where both light gray and semi-translucent samples end up. All in all, the results echo the complexity and unreliability that exists within the topic of petrographic colour.

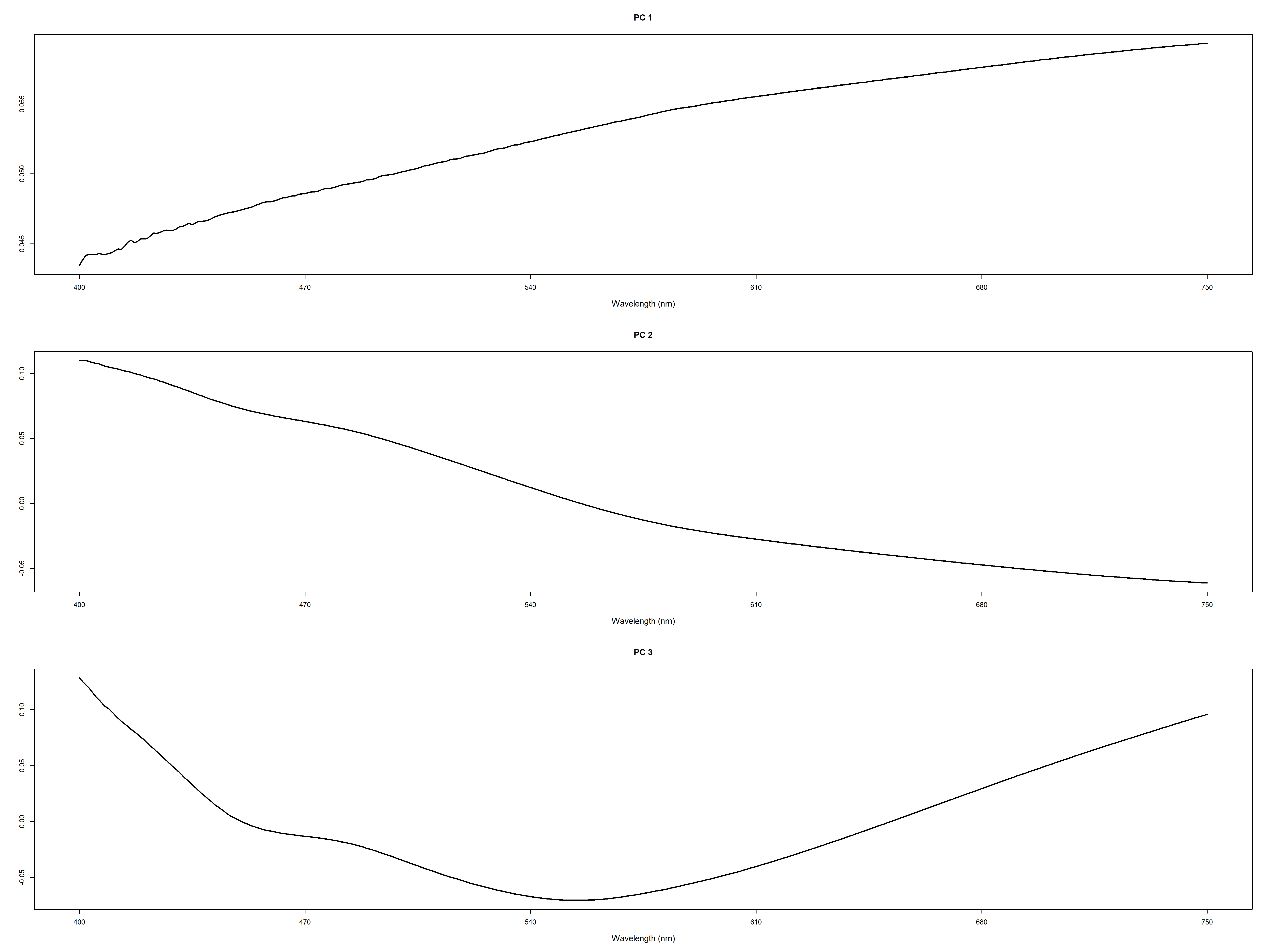


Figure 4.1: Loading lines for PC 1 - 3. The two first components mainly describes features with a linear increase in absorbance across the visual spectrum, but the 3rd component demonstrates more variation, with lower absorbance within the blue-yellow region.

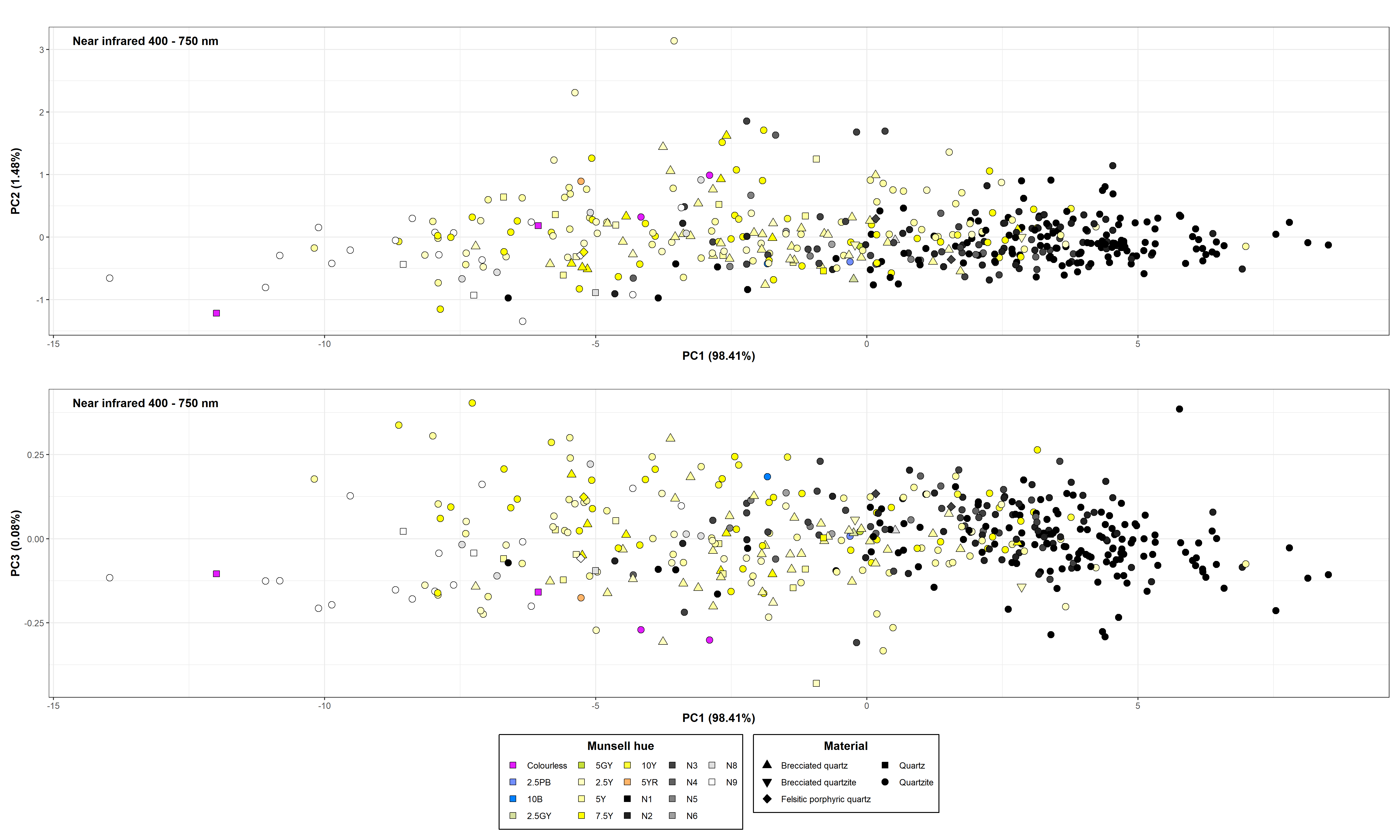


Figure 4.2: PCA score plots for the 400 nm – 750 nm band of the NIR data. Top: PC 1 and PC 2, bottom: PC 1 and PC 3. PC 1 seems to group dark-light material on opposite sides of the plots while PC 2 does little to change the strucure. PC 3 seems to position material with a blue-gray tint towards the top of the score plot.

## 4.2 NIR - near infrared range

In the case of the 1 000 nm - 2 500 nm band of the NIR data, similar features can be observed in the average spectra for all four colour groups, with darker materials exhibiting higher absorbance and the light group the most distinct peaks. The presence of molecular water can be inferred based on the peaks at 1 400 nm and 1 900 nm (Hunt 1977; Sciuto et al. 2019) (fig. 4.3). Peaks also occur at 2 200 nm and 2 350 nm, where the former has been related to combination vibrations of Al-OH and the latter falling within one of the stronger CO3 bands (Clark et al. 1990). Inspection of individual spectra for the light group, however, also reveals a 5th peak for some samples at 2 450 nm. Comparisons to spectra collected on muscovite (Clark et al. 1990, pp. 12 675) shows similar peaks located at 2 200 nm, 2 350 nm, and 2 450 nm. It is therefore possible that the 2 350 nm band in some cases corresponds to CO3 vibrations and in other samples Al-OH, possibly inidicating the presence of muscovite.

The PCA shows a strong trend along PC 1 that seems to correlate with the colour of the material(fig. 4.4). Darker quartzite cluster to the right with white and colourless samples grouping to the opposite side. While the white samples seem to gravitate to the bottom left of the model the samples that are somewhat translucent, such as the brecciated quartz, seem to cluster towards the top-left. At the top of the score plot 3 black samples separate themselves clearly from the rest of the data points. These three samples come from different sites and exhibit no peaks in their spectra, making them an interesting set of outliers.

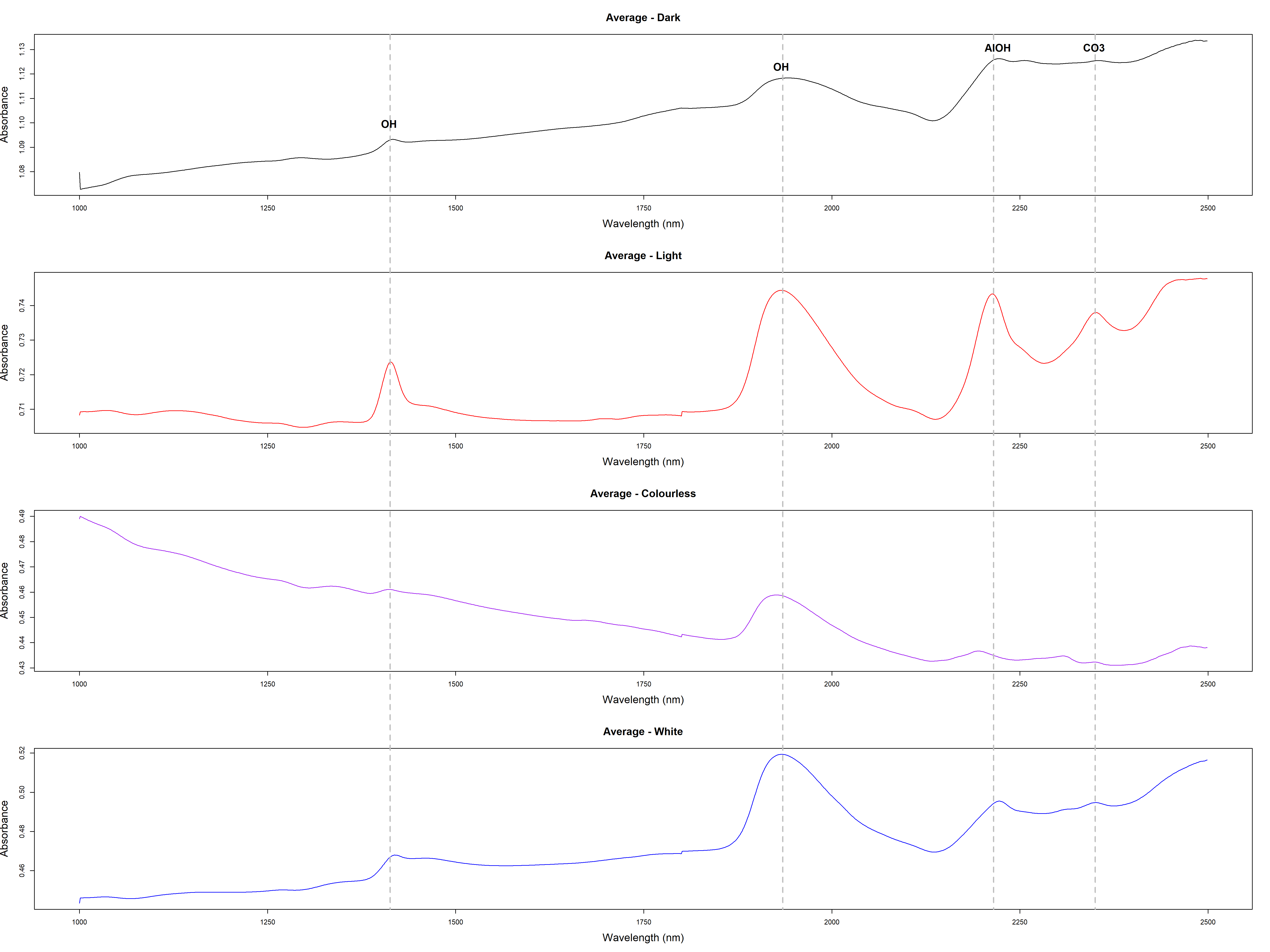


Figure 4.3: Average NIR spectra by hue. Darker samples feature higher overall absorbance, as well as less pronounced peaks, when compared to lighter/colourless samples.

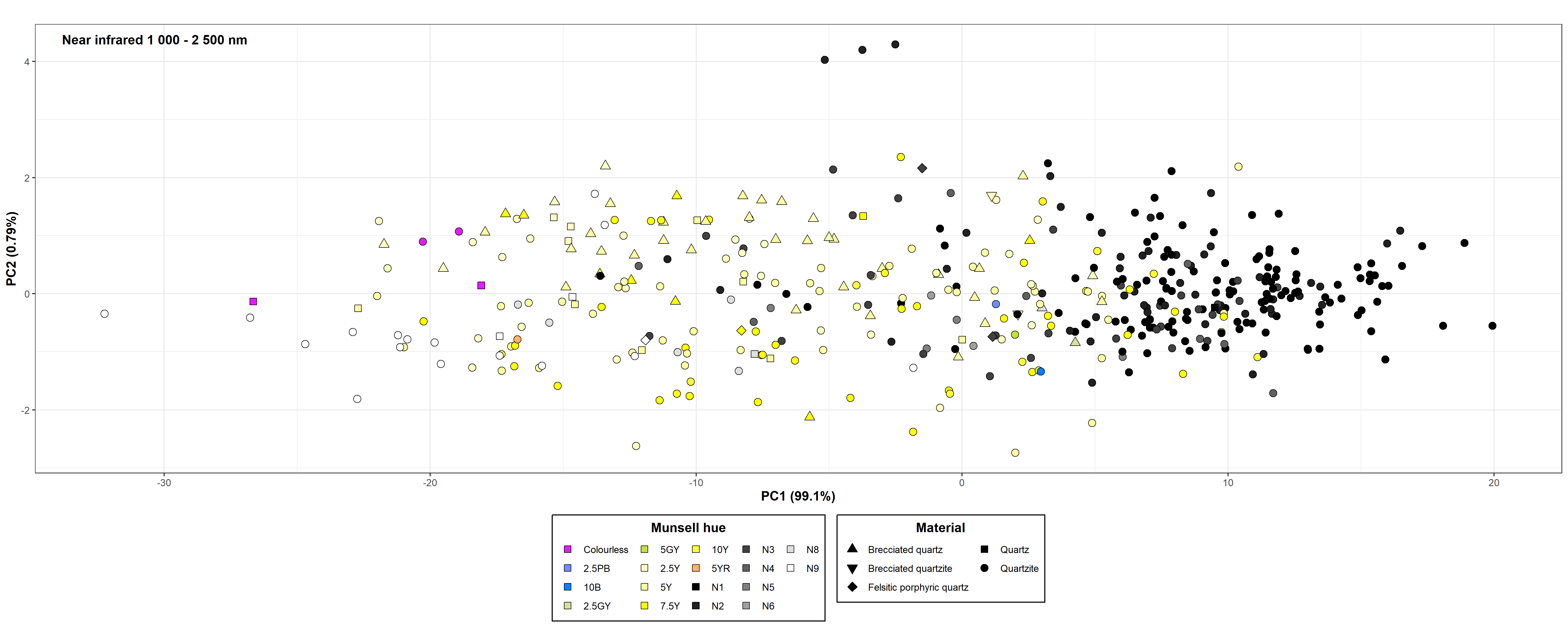


Figure 4.4: PCA score plot of 1st and 2nd components for the 1 000 nm – 2 500 nm region of the spectrum. Quartz-based material with a darker surface group to the right in the model, while those with white surface group in the bottom left. A trend of translucent material and opaque material with a beige tint grouping in the top left can also be seen. Three black samples at the top, from different sites, all feature no distinguishing peaks in their spectra thus separating them from the other samples in the dataset.

## 4.3 Raman

There are two distinctive features present in the Raman data (fig. 4.5). The first are three peaks in the 100 cm-1 - 500 cm-1 band that are typical for SiO2 and quartz-based materials, with the strongest peak occuring at 460 cm-1 (Gillet et al. 1990). These peaks are present for all samples in the dataset. The second feature shows up as two peaks occurring at around 1 287 cm-1 and 1 598 cm-1, and dominate the spectra in terms of intensity for the darker samples. These peak positions fall within what is referred to as the *D* and *G* band respectively, and have been observed in spectroscopic studies of graphite (Cesare and Maineri 1999). The D band occurs as a result of disordered carbon (C) structures whilst the G band is indicative of sp2 carbon networks and caused by stretching in the carbon bonds, which is common in graphite-based materials (Cohen-Ofri et al. 2006; Vollebregt et al. 2012; Lee et al. 2021). Whereas the 2nd peak falls within the typical position of the G band, the position of the D band is commonly identified as being in the 1 330 - 1 350 cm-1 region. It has, however, been documented that the dispersive behavior of the D band can cause a frequency change as a result of the energy of the incident laser (Pimenta et al. 2007, p. 1280). This could thus explain the discrepancy between the observed peak in the dataset and the literature.

A third feature, present in a small subset of the samples, appears as two peaks spaced closely together at ~ 695 cm-1 and 795 cm-1. Comparing these to reference samples from the mineral and rock collections kept at the Department of Ecology and Environmental Science at Umeå University, the same peaks are present in measurements collected on, among others, albite, apatite, diabase and shale. Studies of clay minerals (Kloprogge 2017) have identified a number of band regions in the lower wavenumbers related to Si, Al and OH vibrations, including the 700 - 800 cm-1 band. Considering the petrogenesis of quartzite and the likelihood that clay minerals are present in the material it is possible that this is what generates the feature in the current dataset.

The resulting PCA model (fig. 4.6) shows a clear trend along PC 1 (81.5 %) related to the quartz and D - G peaks. PC 2 and PC 3, however, mainly describes the remaining noise left in the spectra after detrending and preprocessing the data. Interestingly, the loading for PC 4 does include the Al-O-Si peaks around the 700 cm-1 - 800 cm-1 band, which helps in separating out those samples where this feature is present. The measurements with mainly quartz peaks gravitates towards the negative side of PC 1, and those where the graphite peaks are dominant towards the opposite side. It is also clear that the darker material feature more pronounced graphite peaks, whereas the lighter and more colourless objects mainly feature quartz peaks. As in the case of the NIR data, however, there are exceptions that break this trend, as well as a “muddled” middle group with both well-pronounced graphite and quartz peaks. When PC 4 is added the samples with quartz and Al-O-Si peaks gravitate towards the bottom left, separating themselves from the “quarts” group.

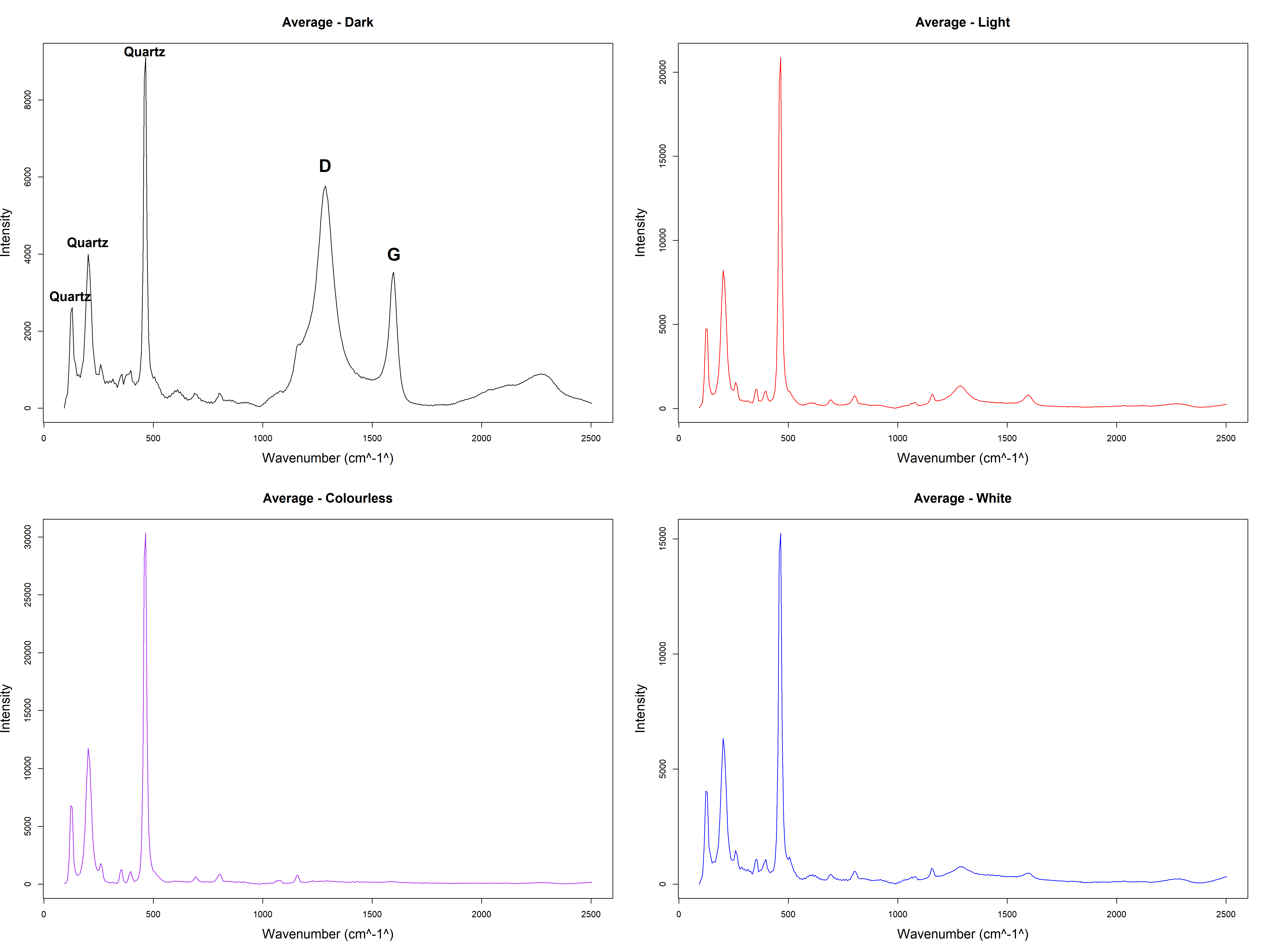


Figure 4.5: Average Raman spectra grouped by their hue. The *quartz peak* at 460 cm-1 is present in all four groups, but the *graphite peaks* at 1 287 cm-1 (D band) and 1 598 cm-1 (G band) mainly appear in the dark group

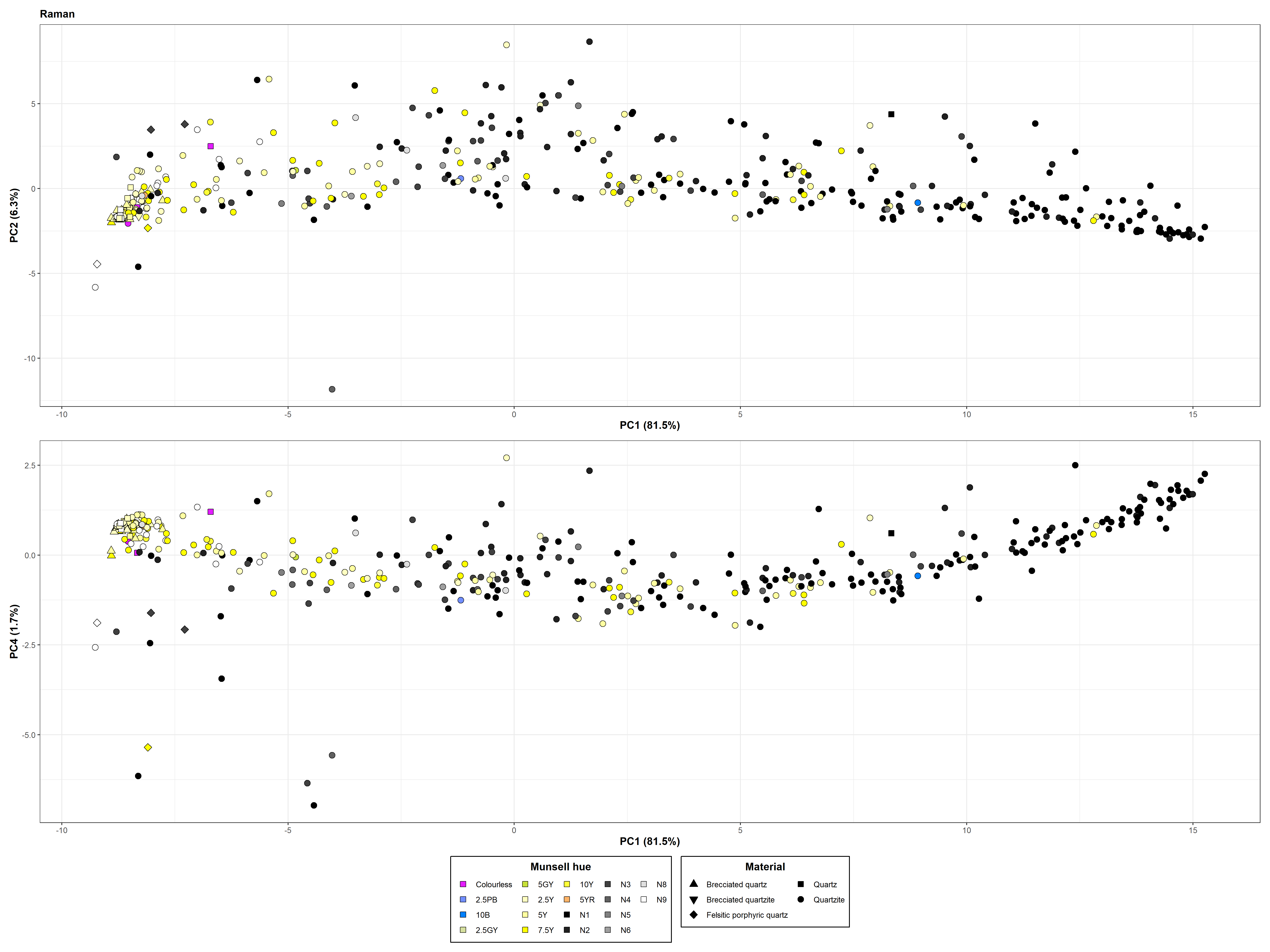


Figure 4.6: PCA score plots for the raman spectra. The main explanatory power can be found in PC 1, which groups the data based on the quartz and D-G bands. As these are the main features in the data, the other components mainly describe the remaining noise, producing the arch-shape in the PCA. PC 4 does, however, also separate out the few samples that feature peaks at 700-800 cm-1 in the bottom left, that may result from Al, Si and OH vibrations.

## 4.4 XRF

Four samples had dimensions that were < 10 mm in size of their smallest dimension, and were therefore excluded from the analysis as there may be complications when comparing these to larger samples (Shackley 2011b, pp. 9–10). The XRF spectrometer is not capable of detecting all of the common elements, and the software therefore calculates a balance (Bal) that represents the remaining unquantifiable elements in a sample. The elements represented in this unquantifiable group are all those with an atomic number lower than 12Mg (REF). Univariate analysis shows that SiO2 makes up the main bulk of the composition of the material, and two major groups can be distinguished based on this content (fig. 4.7). The majority of the readings resulted in a SiO2 content of less than 50%, and about ¼ of the readings (~ 27%) featured a SiO2 content closer to 90%. The “low” SiO2 group also feature a Bal between 40 – 70%.

The two first components of the PCA have an eigenvalue above 1 and the cumulative explained variance is ~ 81.3%. The loadings can be be seen in figure 4.8. In the score plot of PC 1 and PC 2 two large clusters can be seen (fig. 4.9). Interestingly, the smaller cluster on the left side in the score plot includes the samples with a SiO2 content higher than 50%, whilst the larger cluster includes those with lower SiO2 content. It does not seem to be 14Si that is responsible for this clustering, however, but rather 26Fe and 40Zr that are the main contributors. The “low 14Si” cluster shows a trend along PC 2 of quartzite with a darker hue grouping towards the bottom. This may indicate a possible relation to the 26Fe content of the material as 26Fe-bearing minerals such as hematite can contribute to the dark colour in quartzite (Howard 2005, p. 711). The brecciated quartz samples largely group together at the top of the “low 14Si” cluster, with a couple ending up closer towards the “high 14Si” cluster. The white (N9) material is fairly evenly distributed within the right cluster.

The group of measurements that are somewhat separated from the “low 14Si” cluster at the bottom are all but one from the rock painting site of *Finnforsberget*, which is located next to the Skellefte river. The *Finnforsberget* artefacts were sampled twice with object 491 having one measurement closer to the “low 14Si” group and the other the “high 14Si” group with 13Al estimated at 25 %. This artefact was sampled twice on each side, resulting in similar results both times.

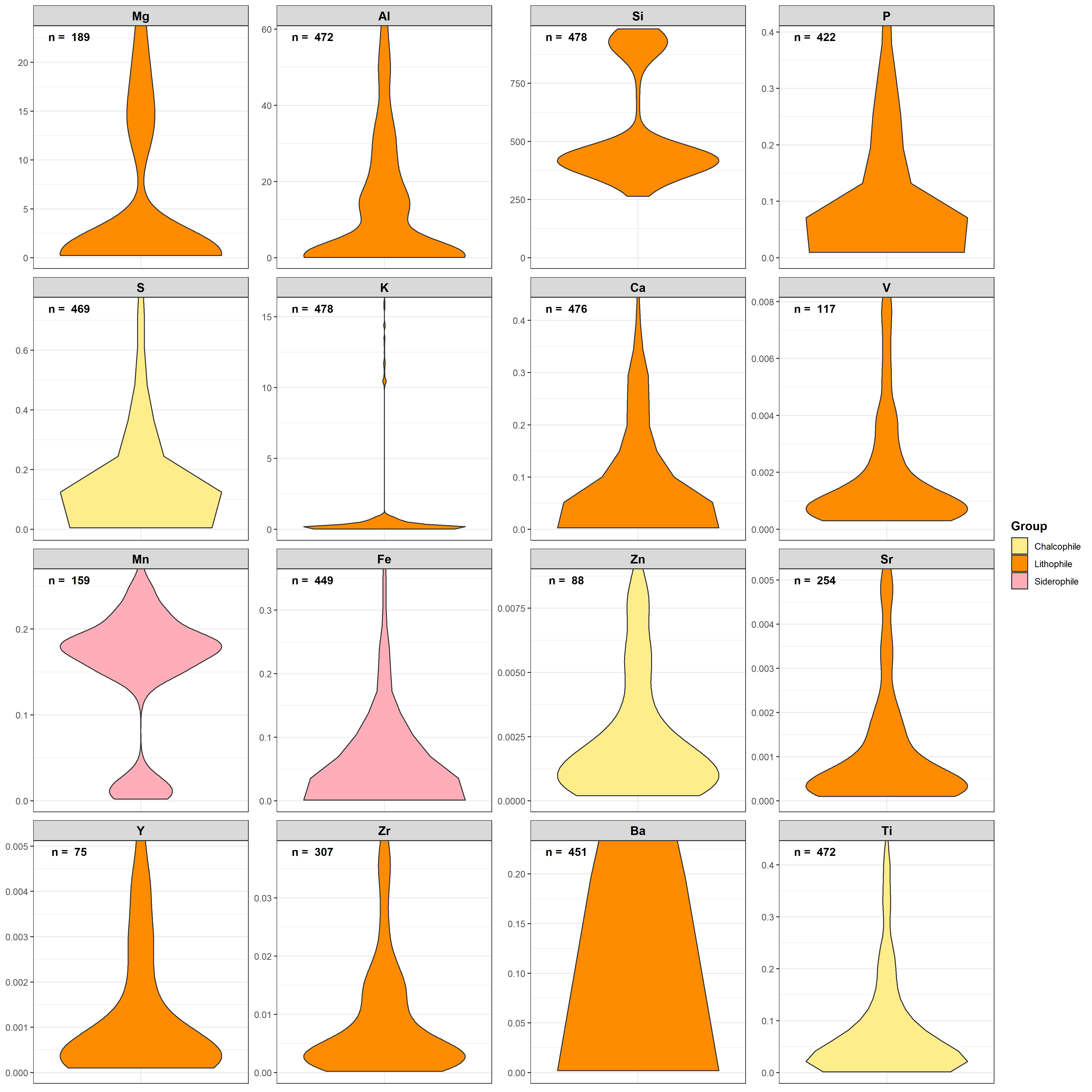


Figure 4.7: Violin plot of percentage element content in quartzite samples measured using pXRF. **n** represents no. of measurements above Limit of Detection (LOD) for that element out of a total of 474 measurements. Note the bimodal distribution of Si measurements, these groups are reflected in the score plot of the PCA

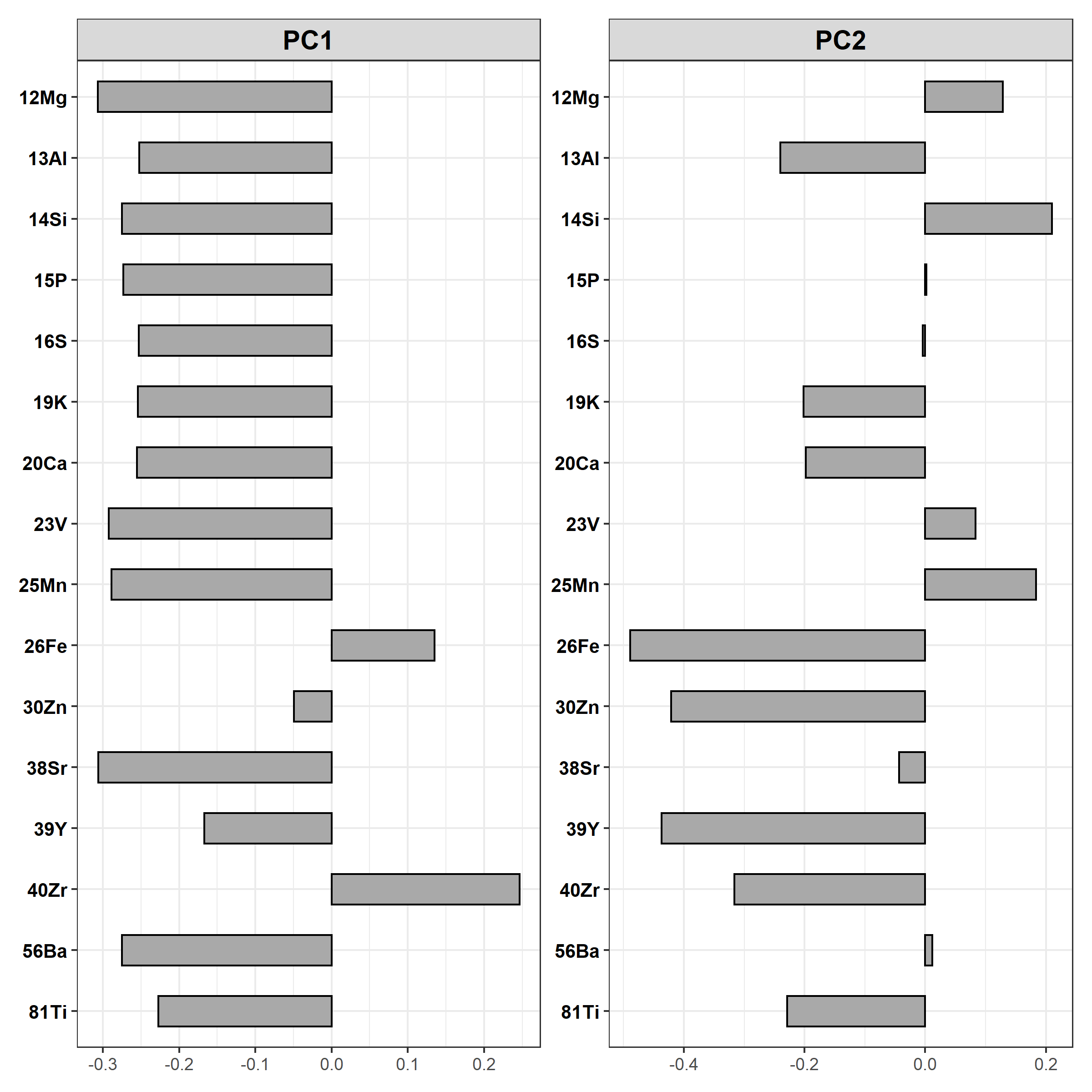


Figure 4.8: Loading vectors (P1-P2) with scores for each variable (element)

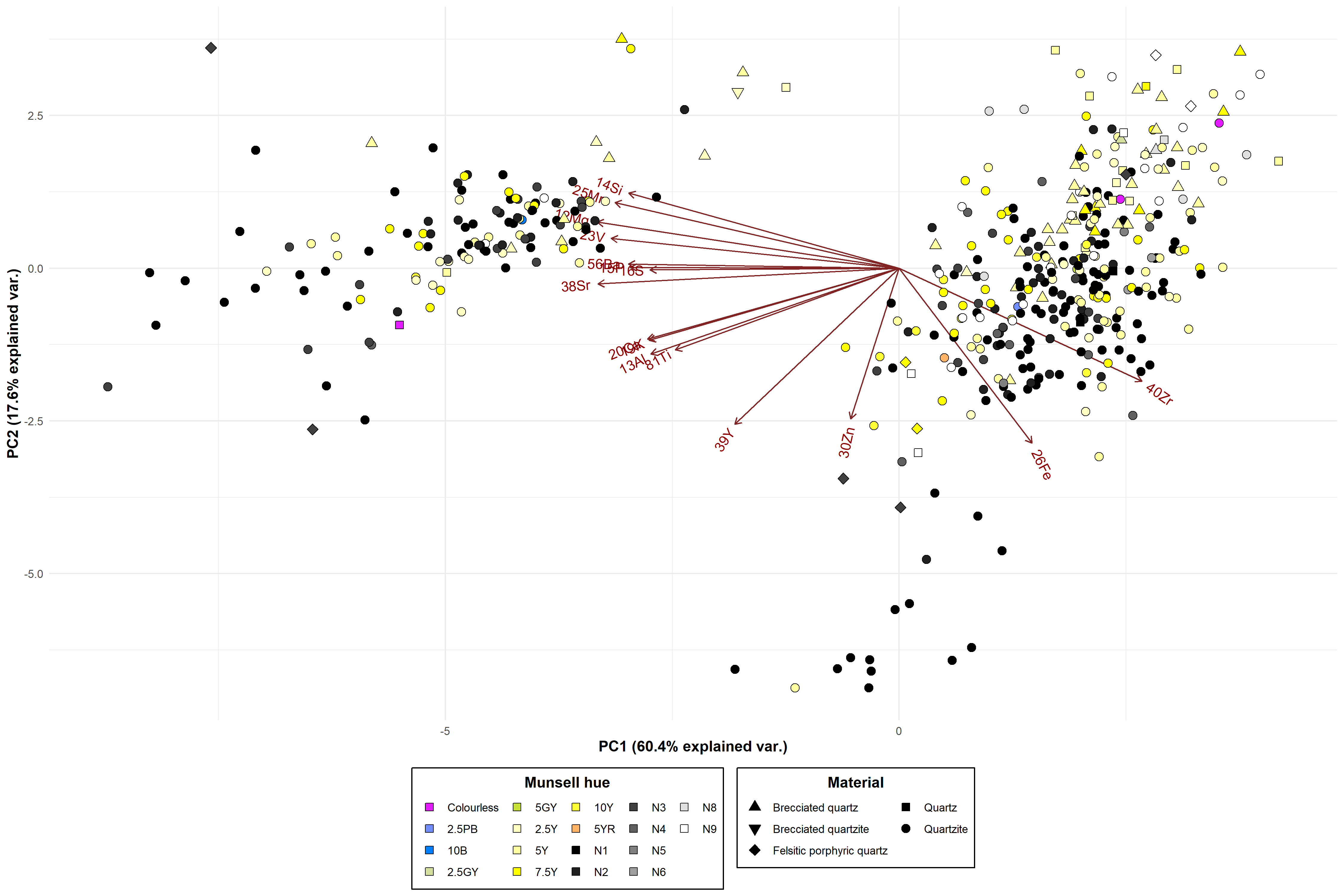


Figure 4.9: XRF score plot of PC 1 and PC 2. The smaller group to the left in the plot includes samples with higher Si content, while the larger group includes samples with Si content < 50%.

# 5 Discussion

The most easily recognizable megascopic feature of petrographic material is its colour, a feature that has been one of the main means of grouping quartzite in North Swedish archaeological research (e.g. Forsberg 1985, pp. 60–64; Käck 2009, pp. 53–55). While it is true that some inference can be made based on colour, such as 26Fe-bearing minerals contributing to a darker colour, this is significantly more difficult with heterogeneous material. Should a quartzite where the matrix is mainly light gray in colour, but streaked with black mineral veins, be considered a *light* or *dark* quartzite? The results above reflects this complexity. While the above models tend to group samples of a similar hue together, especially in the case of NIRS, there are samples that break these trends and thus demonstrate the presence of structural and compositional features that do not necessarily align with the perceived colour of a material. Rather than assigning color based on how it is perceived visually it can be approached in a more systematic manner (e.g. Fairchild 2013). Restricting the NIRS model to the visual range (~ 400 nm - 750 nm) opens up for some inference of color based on the absorbance of light within certain bands. However, as the data was collected using a contact probe the issue of how heterogeneous samples should be treated remains. This issue can be alleviated somewhat with a different sampling approach, for instance using a hyperspectral camera where different pixels can be selected and analysed separately (Sciuto 2019).

In the case of the Raman data the PCA is somewhat lacking in exploratory and descriptive power. Apart from the few samples that feature the 700 cm-1 - 800 cm-1 band peaks the major features in the data are all explained by the first component. As the intensity in Raman spectroscopy is not a direct quantitative indicator this makes it difficult to group materials based on the intensity of the D and G bands, making more of a binary separation of samples with or without *graphite peaks*. That is not to say that it is not possible to attempt quantification using Raman spectroscopy, but rather that this would necessitate a different experimental setup. That said, the presence of the *graphite peaks* may present an important piece of information in the identification of the potential geological origin of the material. Interestingly, mentions of *graphitic quartzite* can be found in an early 20th century description of an excursion to Storfjället, Tärna parish, by the Geological Society (Geologiska Föreningen i Stockholm 1925), but otherwise does not seem to be a frequently used term in the modern day.

The material described as *brecciated quartz* remains relatively grouped together in all of the models, demonstrating some consistency with the original classification. As the samples group with other similarly semi-translucent material, however, it does not seem to distinguish itself enough in the models in order to consider it its own group. This is perhaps not so strange as the original classification by Åhman (1967) refers to the microscopic fragmented configuration of the quartz crystals as being the main distinguishing feature, and while it is possible to discern the presence of SiO structure through non-destructive spectroscopy, there have been no other discernible features related to this group at this stage. It is questionable whether all of the samples in this group truly exhibit brecciation, given the context under which they were collected and classified, which further complicates analysis of this material. Nevertheless, the results of the analysis suggests that there is some in-group variation, regardless of whether the material exhibits breccia.

# 6 Conclusion

The results demonstrate clearly that non-destructive application of all three instrumentations (NIRS, Raman, XRF) are capable of petrographically characterizing some aspect of the quartz and quartzite, however, taken on their own they lack the resolution needed to provide a detailed enough fingerprint for classification. Nevertheless, they do provide a more in-depth understanding of the composition and structure of the material, as well as showcase the complexity in the petrographic character of quartz and quartzite materials. The study represents a good example of a multivariate exploratory approach to the characterization of petrographic material where there also is an absence of reference material. There is a need for methodologies that can be applied in regions with a complex geological setting and vague distribution of potential geological sources. Taking full advantage of the short integration time of these instrumentations in order to build large comprehensive datasets it is possible to discern spectral fingerprints using these methodologies.

# 7 Acknowledgements

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### 8.0.1 Colophon

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#> testthat 3.1.1 2021-12-03 [1] CRAN (R 4.1.2)  
#> tibble \* 3.1.6 2021-11-07 [1] CRAN (R 4.1.2)  
#> tidyr \* 1.1.4 2021-09-27 [1] CRAN (R 4.1.2)  
#> tidyselect 1.1.1 2021-04-30 [1] CRAN (R 4.1.2)  
#> tidyverse \* 1.3.1 2021-04-15 [1] CRAN (R 4.1.2)  
#> tzdb 0.2.0 2021-10-27 [1] CRAN (R 4.1.2)  
#> usethis 2.1.5 2021-12-09 [1] CRAN (R 4.1.2)  
#> utf8 1.2.2 2021-07-24 [1] CRAN (R 4.1.2)  
#> vctrs 0.3.8 2021-04-29 [1] CRAN (R 4.1.2)  
#> viridisLite 0.4.0 2021-04-13 [1] CRAN (R 4.1.2)  
#> withr 2.4.3 2021-11-30 [1] CRAN (R 4.1.2)  
#> xfun 0.29 2021-12-14 [1] CRAN (R 4.1.2)  
#> xml2 1.3.3 2021-11-30 [1] CRAN (R 4.1.2)  
#> xtable 1.8-4 2019-04-21 [1] CRAN (R 4.1.2)  
#> yaml 2.2.1 2020-02-01 [1] CRAN (R 4.1.1)  
#>   
#> [1] C:/Users/sjola/Documents/R/win-library/4.1  
#> [2] C:/Program Files/R/R-4.1.2/library  
#>   
#> ------------------------------------------------------------------------------

The current Git commit details are:

#> Local: main C:/Users/sjola/Documents/VERSION CONTROL/quantifying-quartzite  
#> Remote: main @ origin (https://github.com/MattiasSealander/quantifying-quartzite.git)  
#> Head: [9e8730e] 2022-01-19: updated file name

1. This is not to say that flint was not available or used in these regions [↑](#footnote-ref-21)
2. For comparison, Mjæreum -Mjærum (2012) has put forth a chronology for bifacial points in southwestern Norway [↑](#footnote-ref-24)
3. In this case the majority of the samples from Lycksele are from the rock painting site of Lycksele 301 [↑](#footnote-ref-25)
4. Original Swedish terms are *brecciekvarts*/*brecciekvartsit* [↑](#footnote-ref-28)
5. www.umu.se/forskning/infrastruktur/mal/ [↑](#footnote-ref-30)
6. Raman shifts are typically given in wavenumber (cm-1), instead of spectral wavelength (nm) [↑](#footnote-ref-32)
7. www.slu.se/en/departments/forest-biomaterials-technology/ [↑](#footnote-ref-33)