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A CRITICAL ACCOUNT ON THE AUTOMATED SEM-EDS USAGE IN CERAMIC ANALYSES AT THE EXAMPLE OF PREHISTORIC POTTERY FROM THE SITE OF PETIT-CHASSEUR (3100-1600 BC), SOUTHWESTERN SWITZERLAND

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ABSTRACT

This paper discusses the pros and cons of the application of automated SEM-EDS analysis to the characterization of pottery findings by means of a thorough discussion of the basics of its technology as well as its use in archaeometric research. An in-depth investigation of coarse prehistoric pottery (42 thin sections) from the Petit-Chasseur necropolis (3100-1600 BC, Southwestern Switzerland) provided the perfect testing ground for automated SEM-EDS analysis and resulted in a complete and updated reflection on the capabilities and limitations of this method. An opportunity to produce a quick, reliable, automated, and in-depth petrographic characterization of archaeological ceramics in the form of detailed phase maps stands as a unique feature of automated SEM-EDS technology. Indeed, the information on the composition of aplastic inclusions and clayey groundmass along with the insights on void distribution offer a great resource enabling inferences on raw material choices/provenance and manufacturing technology. However, the present study exposed there are more disadvantages than the ones reported by the literature. A phase identification ignoring crystallographic particularities whatsoever is potentially alarming for mineral sorting, whereas the simplification of lithoclast's internal texture hampers the lithological classification of aplastic inclusions. Notwithstanding listed limitations, the use of automated SEM-EDS in archaeometric research of pottery offers a wealth of useful data which will secure its place in any future investigation of archaeological materials, alongside with the more traditional techniques such as optical petrography, regular scanning electron microscopy and X-ray diffraction.

KEYWORDS: Automated mineralogy, QEMSCAN®, Ceramic characterization, Pottery analysis, Archaeometry, Applied material science, SEM-EDS, X-ray diffraction, Optical microscopy

1. INTRODUCTION

Ceramic findings represent important technological, socio-economic, and chronological indicators (Gliozzo, 2020) as they provide key data to infer on past cultural identities (Gifford, 1960; Peroni, 1967; Shennan and Wilkinson, 2001; Shennan, 2013; Bortoloni, 2016; Manem, 2020), human-environment relationships (Velde and Druc, 1998; Reitz and Shackley, 2012; Michelaki et al., 2015), know-how (Arnold, 1985; Giannichedda, 2006; Knappett et al., 2010; Roux, 2019; Eramo, 2020), and manufacturing traditions (Rice, 1984, 1987; Orton et al., 1993; Skibo and Schiffer, 2008; Levi, 2010; Roux, 2010, 2011; Albero, 2017). Research on clay-based material artefacts has become truly interdisciplinary, integrating natural and social sciences (Rice, 1987; Maniatis, 2002; Cuomo di Caprio, 2007; Levi and Muntoni, 2014; Hunt, 2016; Ion et al., 2016; Liritzis et al., 2020; Michalopoulou et al., 2020; Xanthopoulou et al., 2020). Among several existing methods used for characterization of archaeological pottery, optical microscopy (OM) and scanning electron microscopy (SEM) are, heretofore, the most widely used (Velde and Druc, 1998; Hunt, 2016; Xanthopoulou et al. 2021). Since the first documented application in pottery research in 1883 (Bamps, 1883; Quinn, 2013), OM has progressively been focusing on quantitative data generation. This is due to the impact of emerging computer technology, which ultimately changed the ways in which academics operate (Maritan, 2019). From the first hand-drawn micrographs (Fouque, 1879; Bamps, 1883; Nordenskiöld, 1893) and photomicrographs (Felts, 1942) to the current digitally processed high-resolution images (Carpenito et al., 2009; Dal Sasso et al., 2014; Grifa et al., 2015; Reedy et al., 2017; De Bonis et al., 2020), ceramic petrography has evolved significantly (Maggetti, 2006; Quinn, 2013, 2018; Maritan, 2019). With technological breakthroughs, archaeologists are currently exploring the potential of automatization and machine learning solutions (Papaodysseus, 2012; Sevara et al., 2016; Dietrich et al., 2018; Davis, 2020), which are capable of performing repetitive and time-consuming tasks, thus boosting the data production. In the field of ceramic petrography, several OM- and SEM-based methods were introduced during the last decade. They integrate image analysis applications and automated mineral analysis systems that render data collection quick and repeatable (Aprile et al., 2014, 2019; Albero, 2016; Emami et al., 2016; Hein et al., 2018; Liritzis and Volonakis, 2021). This leads to the acquisition of fully quantitative mineralogical data, which are easy to treat. Systems that attracted attention of the scholars working in the field of pottery analyses are automated SEM-EDS based solutions such as QEMSCAN®. Starting from the pilot study of Knappett et al. (2011), many researchers praised the potential of this tool for quick composition-based classification and inferences on raw material provenance (Knappett et al., 2011; Šegvić et al., 2016b; Cabadas-Báez et al., 2017; Frigolé et al., 2019; Derenne et al., 2020; Carloni et al., 2021; Ogalde et al., 2021). Ten years after the first use of automated SEM-EDS in the characterization of archaeological ceramics, this study aims to thoroughly investigate the pros and cons of its application in pottery studies and to propose a complete and updated reflection on the capabilities and limitations of this method. This is done starting from an earlier work that has applied the automated solution known as QEMSCAN®, OM and conventional SEM-EDS analyses to investigate the petrography and mineralogy of 3rd and 2nd millennium BC pottery from the Petit-Chasseur necropolis (3100-1600 BC, Southwestern Switzerland) (Carloni et al., 2021). Prehistoric ceramics have peculiar features as their paste was frequently prepared by mixing various clays and adding lithoclasts and they were generally fired under low-to-moderate temperatures (< 800 °C) (Arnold, 1985; Velde and Druc, 1998; Prehistoric Ceramic Research Group, 2011; Albero, 2014; Cannavò and Levi, 2018; Javanshah, 2018; Levi et al., 2019; Tanasi et al., 2019). Hence, a thorough petrographic, mineralogical, and micro-textural investigation of aplastic inclusions and clay matrix is crucial for assessing potter's raw material choices and use (Skibo et al., 1989; West, 1992; Hoard et al., 1995; di Pierro and Martineau, 2002; Brunelli et al., 2013; Allegretta et al., 2015; Maritan et al., 2021). Owing to clay matrix heterogeneity and abundance of lithoclasts of various lithology the prehistoric pottery from the Petit-Chasseur necropolis provided the perfect testing ground for automated SEM-EDS technology. Finally, this article intends to contribute to the broader debate on the critical application of automatization and machine learning in archaeological research (Garson, 1990; Georgopoulos, 2016; Traviglia et al., 2016; Anglisano et al., 2020; Davis, 2020).

2. AUTOMATED SEM-EDS: DESCRIPTION OF THE METHOD AND STATE OF THE ART IN CERAMIC ANALYSIS

Automated mineral analysis systems were established in the late nineties for the purposes of the mineral processing industry (Weller et al., 1998; Butcher et al., 2000; Gottlieb et al., 2000; Goodall et al., 2005) and are nowadays largely employed in geological research (Figueroa et al., 2012; Šegvić et al., 2016a, 2018; Benvenuti et al., 2018; Leila et al., 2018; Bell et al., 2020; Kenis et al., 2020; Schulz, 2020; Schulz et al., 2020). They are based on a SEM hardware bundled to an image analysis software and, at present, the most diffused solutions are TIMA-X by TESCAN, Mineralogic Mining by Zeiss, and QEMSCAN® by FEI (now Thermofisher) (Schulz et al., 2020). Other commercial solutions consist of software for automatic mineral identification only, such as AMICS by Bruker as well as INCAMineral and AZtecMineral by Oxford Instruments (Schulz et al., 2020). The analysis is regularly executed on carbon-coated 30 µm-thick sections, but can also be performed on blocks and nonstandard thin sections; the operator can either investigate the entire surface or just a portion of the sample. Possible operating modes are system dependent, but generally include bulk and particle mineral analysis, specific mineral search, trace mineral search, and field/line mapping (Pirrie et al., 2004; Schulz et al., 2020). For analysis of archaeological ceramics, the most applicable mode is the field mapping in which the EDS spectra acquisition moves forward along predetermined fields (Knappett et al., 2011). The stepping interval is predefined and may range between 5 to 20 µm (Knappett et al., 2011; Pirrie et al., 2004) – even though an interval exceeding 5 µm significantly increases the risk of phase misclassification – with a spectra acquisition time of ~10 ms per pixel and a total of hundreds of thousands of spectra acquired for each sample. The software for automatic mineral identification then compares acquired spectra against spectral database and assigns a mineral name to each individual acquisition point, should there be a strong match. This ultimately results in a mineralogical map of the sample entirely based on fully quantitative data with modal mineralogy reported as area % (Fig. 1) (Gottlieb et al., 2000; Pirrie et al., 2004; Knappett et al., 2011; Šegvić et al., 2016b). Whenever an EDS spectrum type cannot be correlated with a library entry the software marks the corresponding area of the map as 'unclassified' (Fig. 1). The share of 'unclassified' can however be reduced by updating the database with user prepared EDS standards. This debugging procedure is particularly useful whenever analyzing complex solid solutions such as the clay minerals.

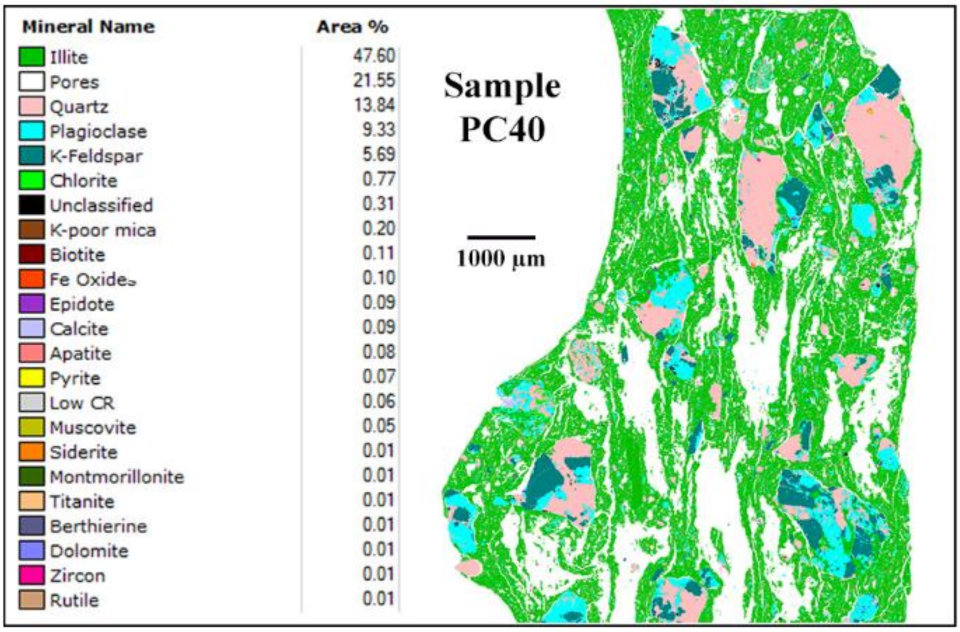

Figure 1. Example of mineralogical map and modal mineralogy resulting from the QEMSCAN® analysis.

During the last two decades, automated SEM-EDS technology has been applied to both archaeological investigation and to several fields of geologic study. It was mostly used as a characterization tool of (a) sediment from sites of archaeological and forensic significance (Pirrie et al., 2009; Campaña et al., 2016; Edwards et al., 2017; Ward et al., 2017; Šegvić et al., 2018), (b) lithic material employed in megalithic architecture (Bevins et al., 2020, 2021), (c) ore deposits and metalworking-related tools (Figueroa et al., 2018; Schulz et al., 2020), (d) ancient cosmetics (Hardy et al., 2006; Hardy and Rollinson, 2021), and (e) ceramics (Knappett et al., 2011; Šegvić et al., 2016b; Cabadas-Báez et al., 2017; Frigolé et al., 2019; Derenne et al., 2020; Carloni et al., 2021; Ogalde et al., 2021). Literature on advantages and disadvantages of the use of automated SEM-EDS technology in the study of archaeological ceramics is summarized in Table 1. Knappett et al. (2011) and Šegvić et al. (2016b) highlighted the accuracy of QEMSCAN® analysis and classification potential of mineralogical maps that combine compositional and textural data. Knappett et al. (2011) for instance refined the petrographic classification by further clustering the Akrotiri jars according to their clay matrix composition and by identifying the areas of the raw material procurement. Šegvić et al. (2016b) successfully characterized clay pellets occurring in the ceramic paste and provided information on non-stoichiometric firing silicates whose composition was later corroborated by microprobe analyses (Borgers et al., 2020). Knappett et al. (2011) further highlighted one important disadvantage, i.e. the misclassification of phases of similar chemistry such as mineral polymorphs. Cabadas-Báez et al. (2017) identified and compared alteration features of volcanic glass particles present in both the Maya ceramics and riverine sediments and, consequently, inferred that volcanic clasts were natural components of the raw material used in pottery manufacturing and not the temper intentionally added by the potter(s). Similarly, Frigolé et al. (2019) underlined the potential of automated modal mineralogy in characterizing the finest aplastic inclusions and groundmass but questioned the ability of automatically identifying lithoclasts. In recent work (2020), QEMSCAN® mineralogical maps were utilized for identification of vessels' primary and secondary forming techniques. The same pottery assemblage was analyzed by Carloni et al. (2021), which put forth the existence of horizontal voids revealer of the use of coiling technology to shape most of studied pots.

Table 1. Literature data on advantages and disadvantages of the use of automated SEM-EDS technology in the study of archaeological ceramics.

		PREVIOUS WORKS						
		Knappett et al., 2011	Šegvić et al., 2016	Cabadas-Báez et al., 2017	Frigolé et al., 2019	Derenne et al., 2020	Carloni et al., 2021	Ogalde et al., 2021
ADVANTAGES	Identical samples may be analyzed by optical microscopy as well as electron microbeam techniques	X	-	-	-	-	-	-
	Operator-independent analysis, highly reproducible and accurate data	X	-	-	-	-	-	-
	Fully quantitative data	X	-	-	-	-	-	-
	Combination of textural and mineralogical data based on elemental spectra	X	X	-	-	-	X	-
	Mineralogical characterization of the finest aplastic inclusions and groundmass	X	X	X	X	-	X	X
	Study of matrix features normally hidden by the dark color generated by the firing reduced atmosphere	X	-	-	-	-	-	-
	Characterization of opaque minerals and (semi)-amorphous phases	X	X	-	-	-	-	-
	Analysis of clay pellets	-	X	-	-	-	-	-
	Automatically generated modal mineralogy	X	X	-	X	-	X	-
	Grain-size distribution of a single phase	-	X	-	-	-	-	-
	Detection of non-stoichiometric firing phases	-	X	-	-	-	-	-
	Characterization of weathering processes affecting discrete phases and aplastic inclusions	-	-	X	-	-	X	-
	Compositional grouping	X	X	-	X	-	X	X
	Identification of phases marking distinct raw material sources	X	X	X	-	-	X	-
	Comparison of ceramics and hypothesized raw material sources	-	-	X	-	-	-	-
	Markers of primary and secondary forming techniques	-	-	-	-	X	X	-
	Different kinds of data obtained all at once	X	-	-	-	-	-	-
DISADVANTAGES	Misclassification of phases of similar chemistry	X	-	-	-	-	-	-
	Polymorphs unclassified	X	-	-	-	-	-	-
	Failure of automated identification of lithoclasts	-	-	-	X	-	-	-

3. CASE STUDY: THE PREHISTORIC POTTERY FROM THE PETIT-CHASSEUR NECROPOLIS

The petrographic study of the Petit-Chasseur ceramics included optical microscopy (OM) and electron microbeam techniques (QEMSCAN®, SEM-EDS) performed on 42 thin sections. The OM analyses were carried out at the Department of Earth Sciences of the University of Geneva using a Leica Leitz DM-RXP polarizing microscope. As per descriptive methods proposed by Whitbread (1989) and Quinn (2013), the main features of matrix, voids, and inclusions were reported serving as a base for the classification of vessel fabric. The whole sample set was analyzed using an FEI QEMSCAN® Quanta 650F apparatus installed at the same university. Measurements were performed on an area of 1.5 x 1.5 cm (fieldscan operating mode; Fig. 2) at a high vacuum, acceleration voltage of 15 kV, and probe current of 10 nA. X-ray spectra acquisition time was 10 ms per pixel, using a point spacing of 5 µm. The sample holder accommodated up to 12 thin sections at the time and measurements lasted ~4 hours per sample. Finally, conventional SEM-EDS investigation was executed on a subset of 8 ceramic thin sections to corroborate QEMSCAN® automated phase interpretation and to characterize the matrix's microtexture. The analyses were carried out at the Microscopy Center of the College of Arts and Sciences of Texas Tech University using a Zeiss Crossbeam 540 apparatus. Data were obtained within a high vacuum environment, using the backscatter detector and acceleration voltage of 15 kV.

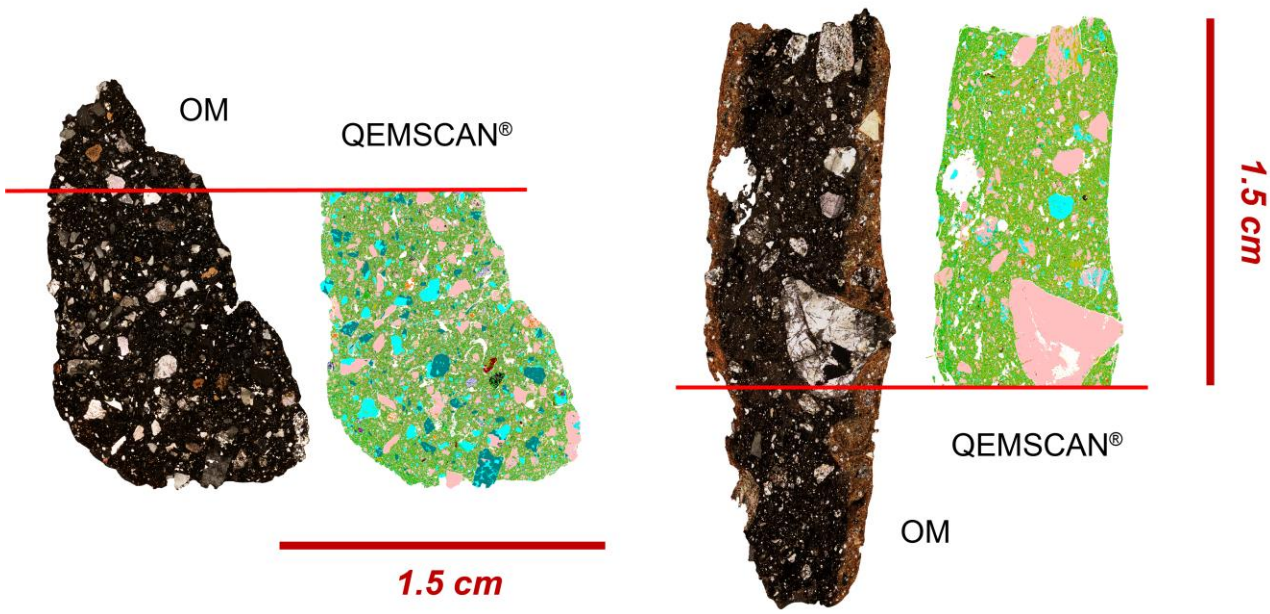

Figure 2. Comparisons of OM and QEMSCAN® measurements made on same thin sections.

Based on OM examination, the Petit-Chasseur pottery displays the characteristics of 9 fabrics (Table 2) (Carloni et al., 2021). The groundmass is optically active, variably colored and associated with different amounts of Fe-rich particles. Voids are vughs, channels, and planar, with random or parallel orientation. Aplastic inclusions largely consist of fragments of magmatic and metamorphic rocks (Carloni et al., 2021). By means of mineralogical maps, QEMSCAN® analysis provided a rapid characterization of the rock and mineral fragments as well as their weathering products present in ceramic paste (Figs. 3 and 4) (Carloni et al., 2021). As indicated earlier the EDS acquisition points in mineralogical maps stand as individual pixels and are colored based on the chart developed by FEI (Schulz et al., 2020). Adjoining inclusions do not appear as individual crystals, but instead as a homogeneous mass. This is a consequence of their identical mineralogy. Subsequently, the internal texture of lithoclasts and rock veins are noticeably simplified in QEMSCAN® maps (Figs. 3 and 4). This may cause difficulties in the classification of rock inclusions as for instance in the case of granite and quartz-feldspar gneiss, which may appear identical (Fig. 3A and B).

Table 2. Aplastic inclusions featuring the 9 fabrics documented in the Petit-Chasseur ceramics. The following abbreviations are used to indicate the grain size distribution (GSD): unimodal (U), bimodal (BI), trimodal (TRI), and polymodal (POLY).

	Fabric	% clasts	Dominant 50-70%	Frequent-Common 15-50%	Few-Rare 0.5-15%	GSD	Max dimension
1	Quartz and feldspar	10%	Quartz	Feldspar, chert	Biotite, white mica	U	0.5 mm
2	Granite	10-20%	Biotite-rich granite, granite	Fine-grained granite with secondary calcite	Quartz, feldspar, biotite, white mica, sedimentary rocks, low-grade metamorphic rocks	BI, TRI	4 mm
3	Fine-grained granite rich in Fe-oxide	10-20%	Fine-grained granite with Fe-oxide	Biotite-rich granite or granite	Quartz, feldspar, biotite, white mica, sedimentary rocks, low-grade metamorphic rocks	BI, TRI	4,5 mm
4	Weathered granite	20%	Weathered granite (secondary calcite)		Quartz, feldspar, biotite, white mica, granite	TRI	6.5 mm
5	Epidote-rich granite	5%	Epidote-rich granite	Quartz	Epidote, feldspar, white mica, Fe-oxide	BI	1 mm
6	Quartz-feldspar gneiss	10-20%	Quartz-feldspar gneiss	Granite	Quartz, feldspar, biotite, white mica, low-grade metamorphic rocks	BI, TRI	6 mm
7	Amphibole gneiss	10-15%	Amphibole gneiss	Granite	Quartz, feldspar, amphibole, biotite, white mica, sedimentary rocks, fine-grained granite	BI, TRI	5.4 mm
8	Glaucophane schist	30%	Glaucophane schist		Epidote, quartz, white mica	POLY	6 mm
9	Calcite	20-25%	Calcite		Quartz, white mica	TRI	2.5 mm

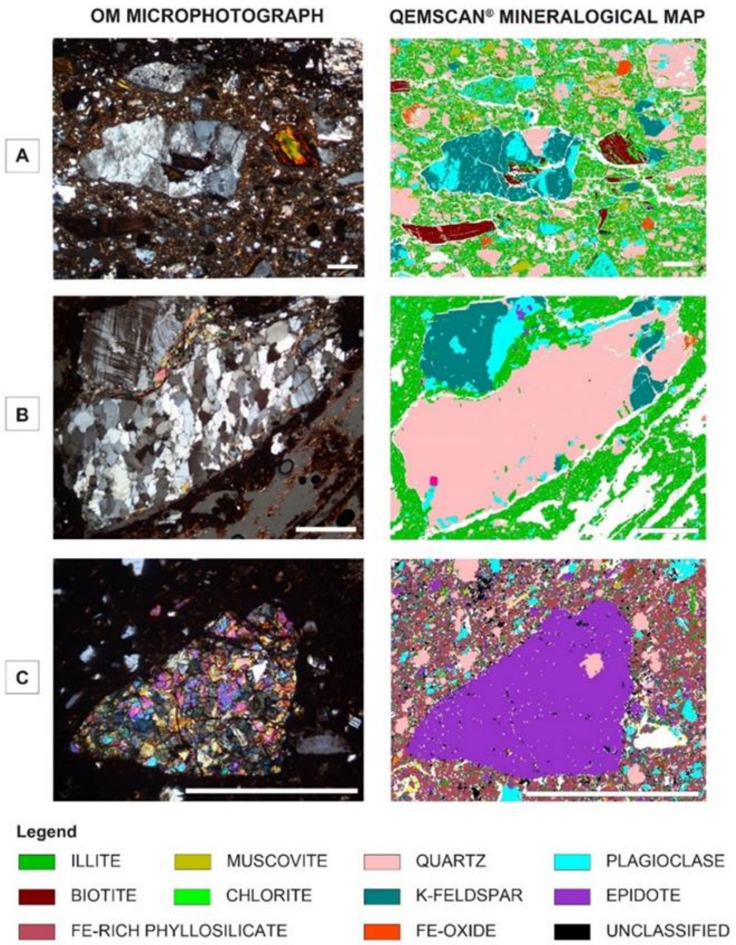

Figure 3. Comparison between optical microphotographs (cross-polarized light) and QEMSCAN® mineralogical maps of selected aplastic inclusions: A) biotite-rich granite; B) quartz-feldspar gneiss; C) epidote vein. Width of the white bar: 500 µm.

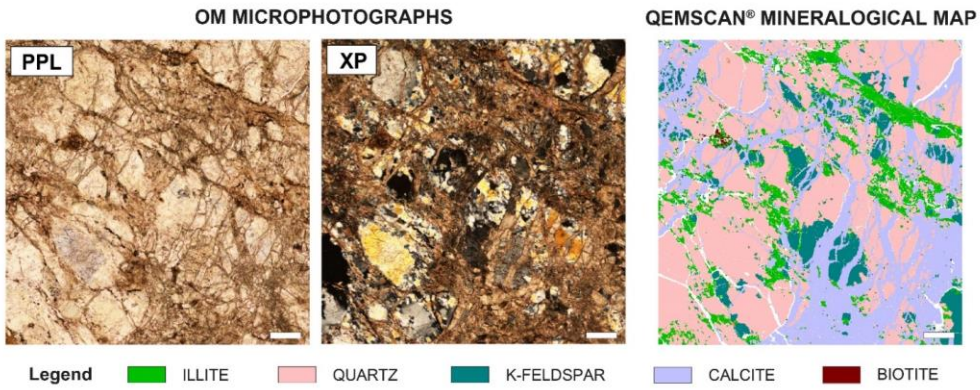

Figure 4. Comparison between optical microphotographs in plane-polarized light (PPL) and cross-polarized light (XP) and QEMSCAN® mineralogical map of a weathered granite. The examined intrusive rock is highly altered by infillings of secondary calcite. Width of the white bar: 500 μm.

A modal mineralogy of analyzed ceramics may be calculated using area percentages (area %) of each identified phase (Fig. 1). Each mineralogical map was thoroughly studied and samples displaying the same OM fabric characteristics showed similar modal mineralogies. Obtained modal mineralogy supported the classification carried out by OM (Fig. 5). However, it should be noted that fabrics 2, 3, and 6 show very similar modal mineralogy and only the OM study allowed identification of various rock inclusions – i.e. granite, microgranite, and quartz-feldspar gneiss (Carloni et al., 2021). With regard to the distribution of aplastic inclusions QEMSCAN® data cannot be used for regular morphometric analysis commonly required by ceramic petrography (Quinn, 2013; Dal Sasso et al., 2014). The reason should be sought in the data format produced by the image analysis software. Firstly, the software breaks the analyzed sample area into 'particles' based on the recognition of voids or empty spaces. Therefore, a lithoclast or a discrete phase present in ceramic paste may be recognized as an individual particle if it is surrounded by a void (Fig. 6A). However, if there is no empty area separating the lithoclast/discrete phase and ceramic groundmass, the former is not individualized and considered as single particle (Fig. 6B). If voids are sparse and minute, the system may identify the entire ceramic fragment as one large particle, which is common in rock analysis (Zhang et al., 2021). Secondly, the software classifies various phases of a particle as 'grains' (Fig. 6). Grains do not define single crystals observable in OM which, as such, are not individually represented in the mineralogical map (Figs. 3C and 6A). It should be said that the software allows to treat the 'particle' and 'grains' in many different ways and offers the opportunity to perform a variety of morphometric studies. However, none of these can automatically reproduce the regular grain size distribution carried out by ceramic petrographers to shed light on the original characteristics of the raw material and eventually recognize addition of aplastic material by the potter.

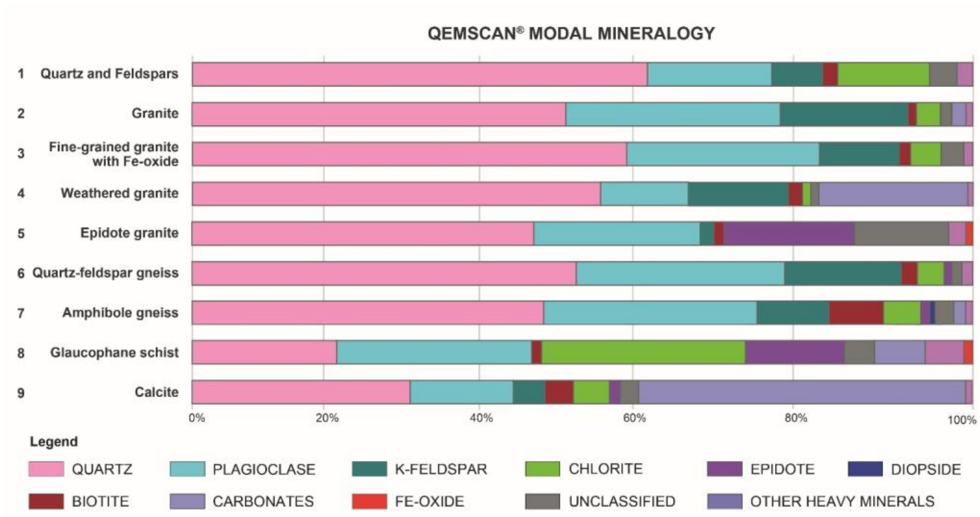

Figure 5. QEMSCAN® modal mineralogy of clasts for each fabric.

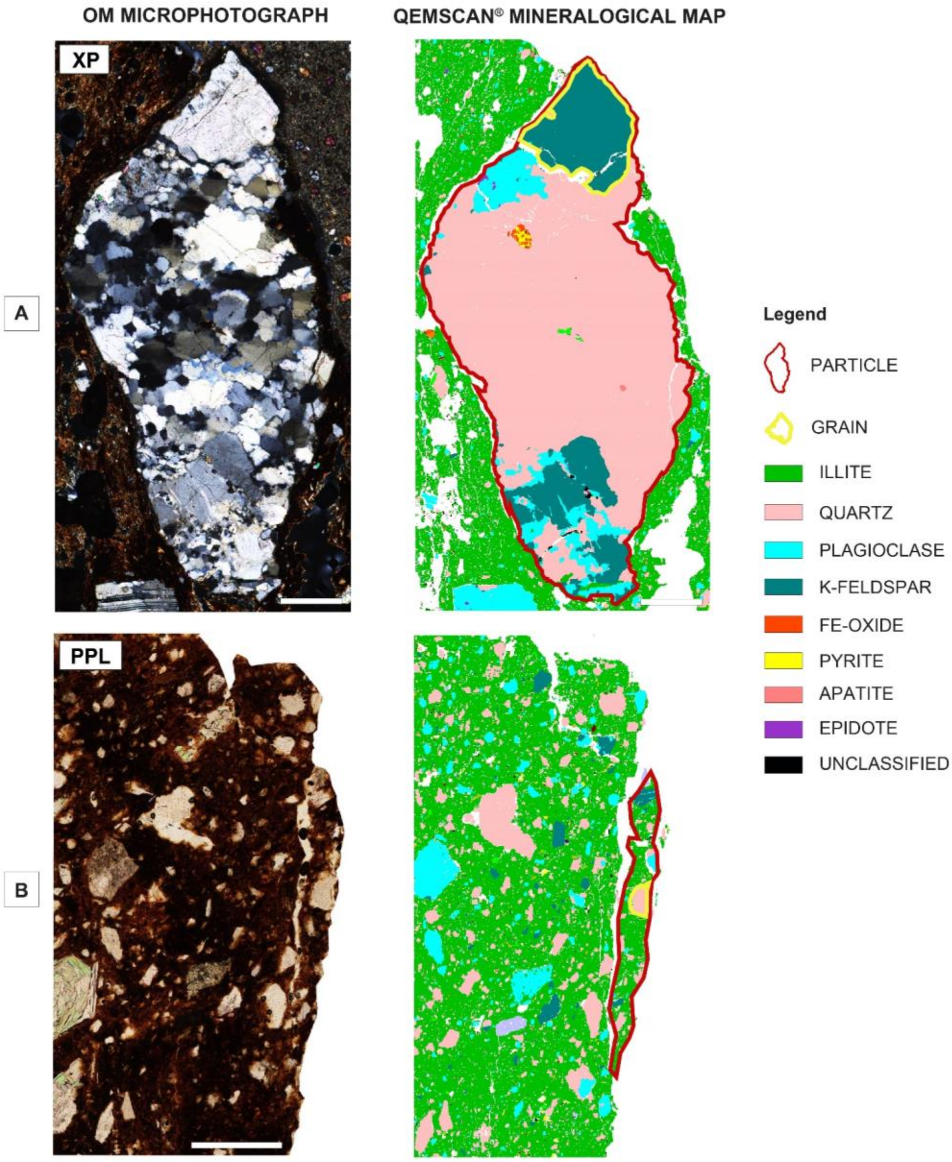

Figure 6. Examples of sample areas classified as 'particles' and 'grains' by iExplorer depending on the presence/absence of voids: A) quartz and feldspar gneiss inclusion in sample PC40; B) portion of ceramic paste of sample PC75. Width of the white bar: 500 µm.

The QEMSCAN® analysis was additionally used to infer on the composition of the matrix of the Petit-Chasseur's ceramics. X-ray diffraction analysis disclosed presence of 10Å phyllosilicates (Carloni et al., 2021). Automated SEM-EDS analysis showed that 5 types of sheet silicates were documented in the matrix (Fig. 7): 1) illite; 2) illite and muscovite; 3) muscovite, illite, K-poor mica (<5% K_2O); 4) Fe-rich phyllosilicate (<20% Fe_2O_3); 5) illite coupled with an unclassified phase. A characteristic Si/Al ratio was used to differentiate illite and K-poor mica. The matrix composition revealed by QEMSCAN® was later corroborated by SEM-EDS analysis (Fig. 8; Tables 3 and 4) (Carloni et al., 2021). In addition, the SEM-EDS analysis allowed to precisely identify the composition of Fe-rich phyllosilicate as Al-rich stilpnomelane (Fig. 8; Table 3) (Carloni et al., 2021) which constitutes the micromass of sample PC73 (Fig. 7D). There are however three major differences when it comes to the findings of automated and standard SEM-EDS analyses. First off, the QEMSCAN® characterized the matrix of PC32 as illite-based (Fig. 7E), whereas SEM-EDS examination revealed its composition of vermiculitized mica (Fig. 8; Table 3). Secondly, in the matrices that are heterogeneous the relative abundance of different clay minerals provided by two methods does not match (Table 4). For instance, according to the software for automatic mineral identification sample PC63 has low rates of K-poor mica (Fig. 7C), whereas regular SEM-EDS analyses showed K-poor mica being the major constituent of ceramic groundmass. Thirdly, the presence of mixed-layer phases (e.g. illite-smectite) was not detected by QEMSCAN® and alteration phenomena (e.g. illitization) were not precisely characterized (Table 4). Thus, sample PC05's groundmass has largely been classified as illitic and, in minor amounts, muscovitic (Fig. 7B), whereas the observation of crystal shape/size documented via BSE images and EDS phase chemistry revealed that illite reported in the mineralogical map of PC05 is actually an altered muscovite (Fig. 8; Table 3).

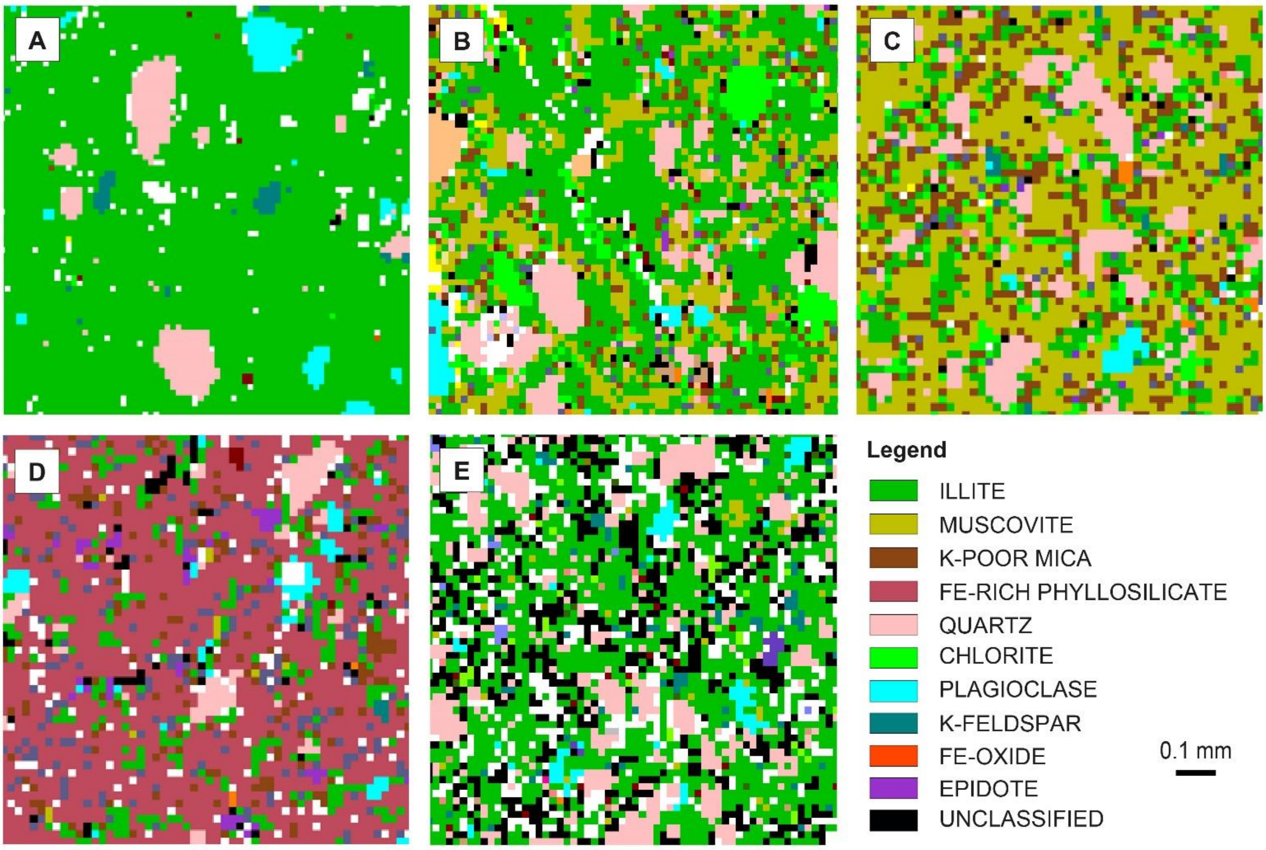

Figure 7. Typical clay matrices of the Petit-Chasseur ceramics according to the QEMSCAN® mineralogical maps: A) illite (sample PC56); B) illite and muscovite (sample PC05); C) muscovite, illite, K-poor mica (sample PC63); D) Fe-rich phyllosilicate (sample PC73); E) illite coupled with an unclassified phase (sample PC32).

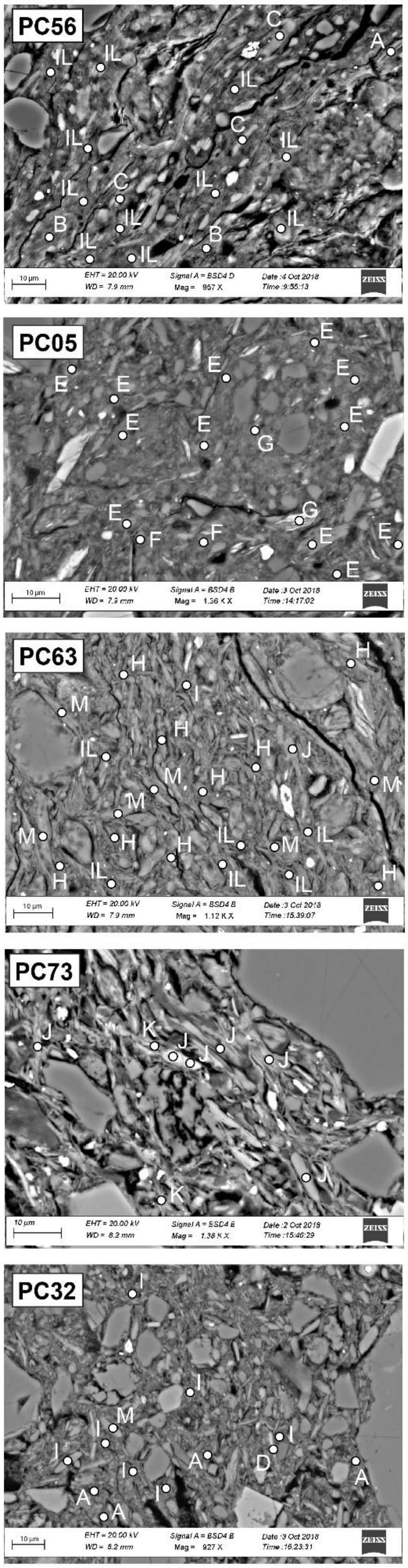

Figure 8. Backscattered image of typical clay matrices of the Petit-Chasseur ceramics: samples PC56, PC05, PC63, PC73, PC32. Exemplary EDS spectra (A, B, C, IL, etc.) may be found in Table 3.

Table 3. Exemplary EDS spectra of the ceramic matrices illustrated in Figure 8.

Sample	Mineral	Spectrum	SiO_2	Al_2O_3	K_2O	FeO	MgO	CaO	Na_2O	TiO_2	SO	P_2O_5	Total
PC56	Illite (IL)	568	54.0	27.4	6.5	5.9	4.6	1.1	-	0.6	-	-	100.1%
	Illite (IL)	555	57.6	24.8	5.1	5.5	5.4	1.3	-	0.3	-	-	100.0%
	Illite-smectite (A)	571	46.5	20.5	3.1	14.3	13.4	1.3	-	0.9	-	-	100.0%
	Chlorite (B)	564	49.7	25.6	2.3	2.7	18.1	1.6	-	-	-	-	100.0%
	Weathered mica (C)	561	53.1	27.4	8.7	5.7	4.1	-	-	0.9	-	-	99.9%
PC05	Muscovite (illitization) (E)	367	49.6	32.6	6.5	6.5	1.2	1.9	0.5	0.5	0.6	-	99.9%
	Muscovite (illitization) (E)	382	52.0	29.8	9.3	6.6	2.3	-	-	-	-	-	100%
	Illite (alteration product of muscovite) (F)	371	60.4	24.4	5.2	4.6	1.1	1.2	0.4	2.3	0.3	-	99.9%
	Chamosite (G)	368	29.5	27.8	1.4	34.1	6.8	0.5	-	-	-	-	100.1%
PC63	K-poor mica (H)	454	54.7	30.5	4.6	4.9	1.2	2.1	0.4	1.6	-	-	100%
	Muscovite (M)	451	50.6	37.2	9.2	1.8	0.8	-	0.5	-	-	-	100.1%
	Illite (IL)	462	65.4	23.2	3.8	4.7	0.9	1.7	0.3	-	-	-	100%
PC73	Al-rich stilpnomelane (J)	159	50.8	39.6	0.6	4.2	1.8	2.3	-	-	0.3	0.4	100%
	Al-rich stilpnomelane (J)	171	42.3	32.3	0.8	20.4	1.6	1.8	0.3	0.5	-	-	100%
	Muscovite (smectitization) (K)	166	54.8	29.7	0.7	10.8	2.1	1.5	-	0.4	-	-	100%
	Muscovite (smectitization) (K)	170	73.7	15.8	0.3	7.0	1.1	1.3	-	0.9	-	-	100%
PC32	Vermiculitized mica (I)	487	55.7	20.1	5.1	4.8	12.4	1.3	-	0.7	-	-	100.1%
	Vermiculitized mica (I)	493	58.0	18.5	5.8	4.7	10.5	2.1	-	0.4	-	-	100%
	Muscovite (M)	500	51.0	35.0	10.1	2.0	1.4	-	0.5	-	-	-	100%

Table 4. Comparison between the QEMSCAN®- and SEM-EDS-based classification of typical clay matrices of the Petit-Chasseur pottery. Listed phases are ordered according to their relative abundance, from the greater to the lesser.

Sample	QEMSCAN®	SEM-EDS
PC56	Illite	Illite and illite-smectite
PC05	Illite and muscovite	Muscovite (illitization) and illite (alteration product of muscovite)
PC63	Muscovite, illite, K-poor mica	K-poor mica, muscovite, illite
PC73	Fe-rich phyllosilicate	Al-rich stilpnomelane
PC32	Illite coupled with an unclassified phase	Vermiculitized mica

From a textural point of view, the QEMSCAN® mineralogical maps permit one to study the matrix features generally masked by the dark color resulting from a reduced firing atmosphere, e.g. the homogeneity/heterogeneity (Quinn, 2013). This was the case for the groundmass of PC05, which appeared homogeneous in OM and heterogeneous in SEM BSE images (Fig. 9). The dark color of the matrix also hampered the observation of opaque minerals, thus failing to provide evidence on Fe-oxide inclusions which were recognized solely based on QEMSCAN® imagery (Fig. 9). In addition, Fe-rich particles were observed by OM in most of analyzed ceramics while the QEMSCAN® investigation allowed their characterization and semi quantification (Carloni et al., 2021). Iron-bearing particles are composed of Fe-oxide, siderite, and ilmenite and their area % greatly vary from one fabric to another (Fig. 10). The quantification of the Fe-rich particles relative abundance and observation of their spatial distribution suggested that that Bell Beaker potters were particularly interested in Fe-rich raw material capable of generating red color of ceramic vessels via an oxidizing firing atmosphere (Carloni et al., 2021).

The void distribution revealed by QEMSCAN® allowed further insights in shaping and decoration techniques (Carloni et al., 2021). The presence of horizontal voids is a remnant of juxtaposition of various elements used to build the form of the pot (Fig. 11A), whereas elongated voids oriented parallel to the vessel's margins likely resulted from beating practice (Fig. 11B). Finally, sherds bearing cordons display discontinuities in the ceramic texture in the area where plastic decoration was applied (Fig. 11C). These inferences on pottery forming technology are in line with the independent study carried out by Derenne et al. (2020).

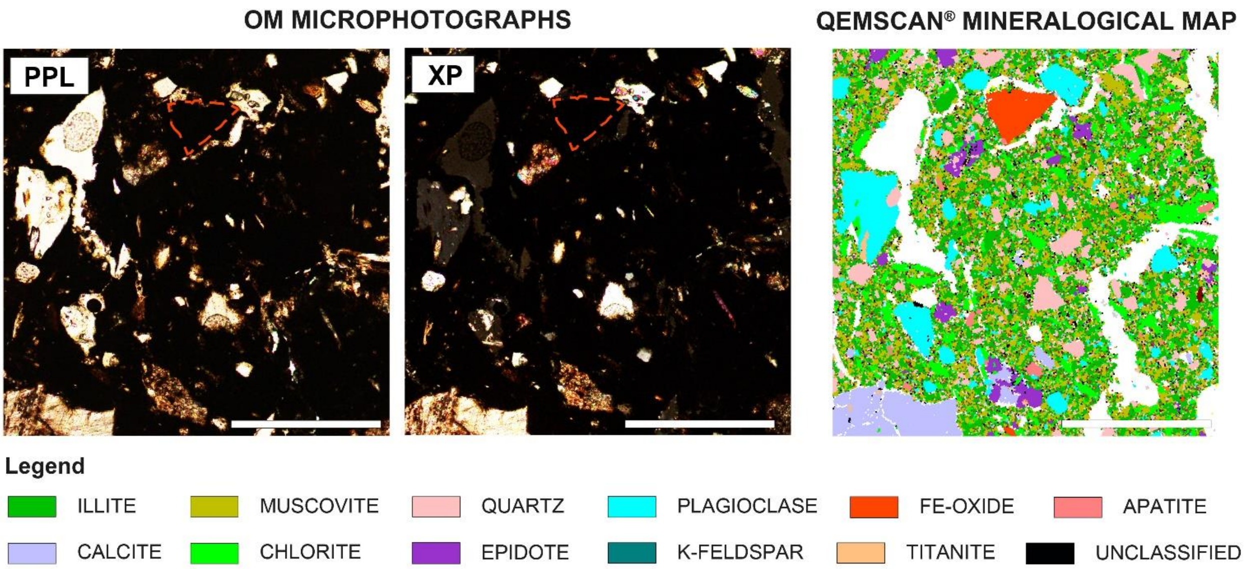

Figure 9. Comparison between optical microphotographs and QEMSCAN® mineralogical map of the matrix of PC05. Width of the white bar: 500 μm.

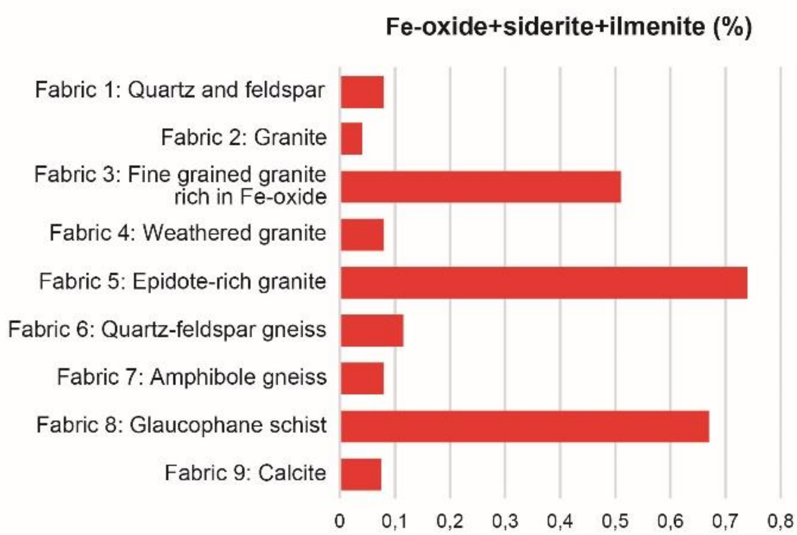

Figure 10. QEMSCAN® quantification of total Fe-oxide, siderite, and ilmenite by fabric. Values expressed as area percentage (%).

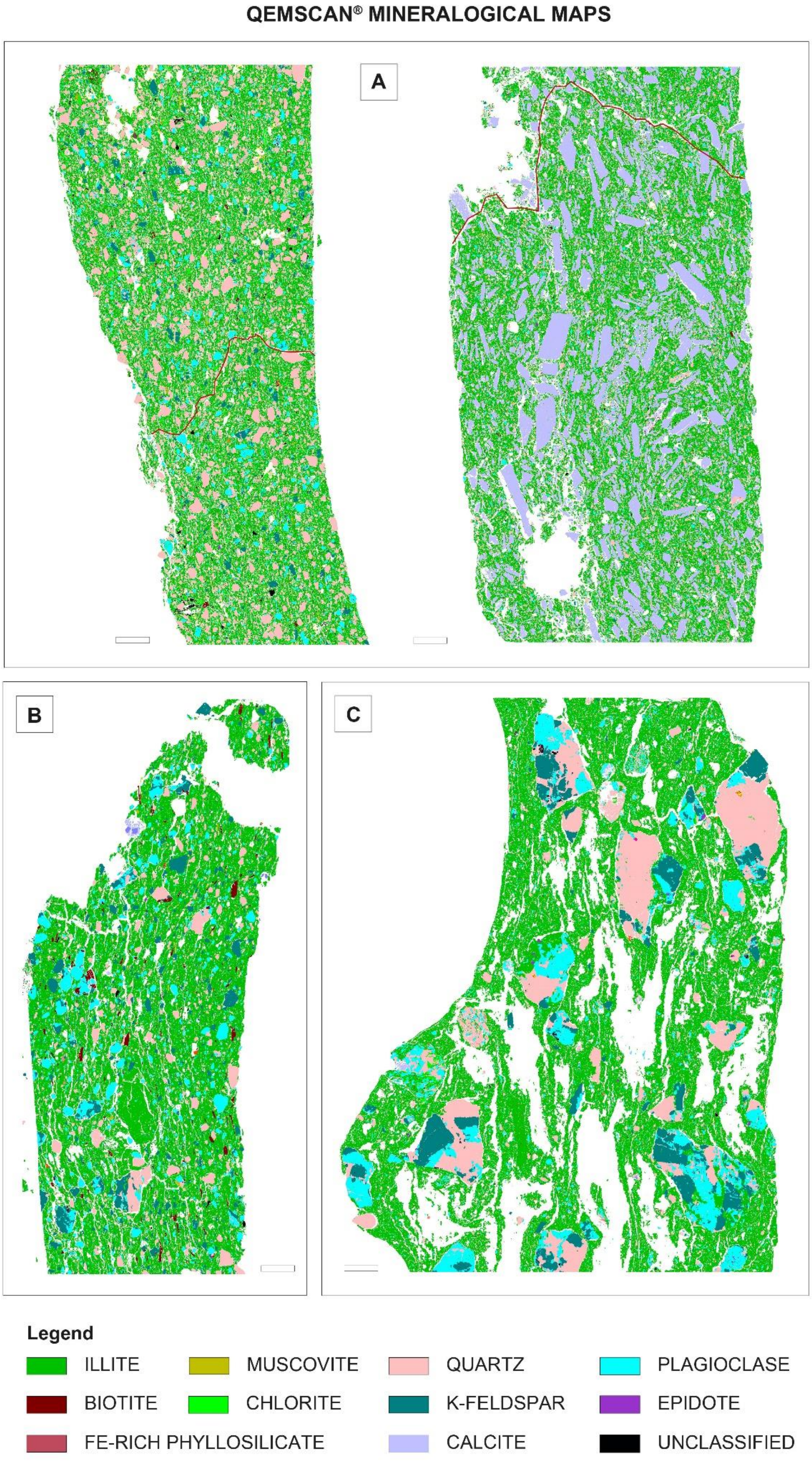

Figure 11. Void distributions as evidence of shaping and decoration techniques in the QEMSCAN® mineralogical maps: A) horizontal voids indicating the use of the coiling as primary forming technique; B) elongated voids oriented parallel to the vessel's margins pointing out the performing of beating as secondary forming technique; C) discontinuities in the ceramic matrix revealing the application of plastic decoration.

4. DISCUSSION: ADVANTAGES AND DISADVANTAGES OF QEMSCAN® ANALYSIS

The research on the Petit-Chasseur's pottery provided the perfect testing ground for the capabilities and limitations of automated SEM-EDS technology in archaeological inquiry (Table 1; section 2). The experience made with the prehistoric pottery of the Petit-Chasseur necropolis highlighted the main advantage of the method consists in the possibility of obtaining a quick, automatic, and in-depth material characterization in the time frame of a few hours per sample. Multi-domain archaeological research also points out the advantages of automatization (Mark Raab, 1993; Huhtamo, 1999; Knappett et al., 2011; Tal, 2014; Georgopoulos, 2016; Sevara et al., 2016; Traviglia et al., 2016; Davis, 2019, 2020; Bickler, 2021) and represents a significant evolution from the first application of computer-based methods in the field of archaeology (Cowgill, 1967; Whallon, 1972; Wilcock, 1973). In the case of QEMSCAN® research, this study made apparent that the operator intervention is pivotal in the refinement of acquired data when dealing with materials of peculiar composition where regular, conventional classification methods are not sufficient (sections 2 and 3). Therefore, the EDS spectra database (section 2) had to be further developed, which requires an in-depth knowledge of mineralogy and phase chemistry. This ultimately makes a debugging procedure suitable only for trained specialists.

The fact that mineral identification relies heavily on phase chemistry and not optical properties (section 2) reduces the risk of misclassification of discrete phases, individual crystals rock composition, and alteration products (Figs. 3 and 4). As always, the procedure of mineral identification is not without problems. Knappett et al. (2011) previously outlined that minerals of similar chemical compositions are commonly mischaracterized. This is due to the differences between them being primarily crystallographic in nature and can be identified more accurately through their optical and diffraction properties. Hence, what can be easily characterized by OM may result in a very complex and long debugging procedure, where successful outcomes are not certain. With regard to the analysis of lithoclasts, the texture simplification through software manipulation became risky as rocks may be of similar composition and contrasting origin and nature (e.g. magmatic vs. metamorphic; section 3). In the case of coarse ceramics, this caused difficulties whenever grouping the pottery according to their modal mineralogy (Fig. 5) and presented evidence that the automated SEM-EDS technology cannot entirely replace the OM in the study of ceramics, at least not in its present form. Other encountered limitations include inability to automatically measure the grain size distribution of aplastic inclusions in ceramic paste. As a matter of fact, the procedure in which the software collects and processes the data does not allow for automatic recognition of lithoclasts as highlighted by Frigolé et al. (2019) and this work found it also hampers any proper morphometric study of them (Fig. 6; section 3). This problem can be mitigated by treating BSE images by means of digital image analysis as suggested by Dal Sasso et al. (2014), Aprile et al. (2019), and Maritan (2019), which would provide a semi-automatic grain size distribution of examined ceramic pastes. It should be said however that the problem of the grain size distribution analyses of aplastic inclusion does not truly exist in case of fine-grained (historical) ceramics bearing monomineralic inclusions. Šegvić et al. (2016b) succeeded in the analysis of morphometric features of quartz and feldspar inclusions in Hellenistic potsherds from the Adriatic settlement of Issa. Nevertheless, the shortcomings of automated SEM-EDS technology in morphometric analysis are offset by modal mineralogy, allowing for compositional properties of analyzed pottery and use of different raw materials to be easily and quickly recognized (Fig. 5; section 3). In addition, one can obtain a modal mineralogy for just one phase or a selection of minerals and observe the difference in that regard throughout the sample set (Fig. 10).

Concerning the analysis of ceramic matrix, the QEMSCAN® system recognized the use of distinct types of raw clays (Fig. 7) and thus enhanced the OM-based grouping (Carloni et al., 2021), taking into account the texture and optical behavior of the matrix, but not its mineralogy (Rice, 1987; Velde and Druc, 1998; Cuomo di Caprio, 2007; Levi, 2010; Quinn, 2013). The case study of the Petit-Chasseur ceramics therefore demonstrated that mineralogical maps provide a powerful base to qualitatively discriminate ceramic matrices. It is true that, in some cases, the classification of clay minerals was not highly accurate (Table 4) but revealed similarities and differences among the matrices of the sample set. The classification of altered muscovite as illite in the micromass of sample PC05 (Figs. 7B and 8; Tables 3 and 4) is not false owing to EDS chemistry of the former being very similar to that of the latter (Carloni et al., 2021). The presence of a K-poor micaceous phase in the matrix of sample PC63 (Fig. 7C) was corroborated by SEM-EDS analysis (Fig. 8; Table 3) as well as the occurrence of Fe-rich phyllosilicate in sample PC73 (Figs. 7D and 8; Table 3). Individual spectra collected from peculiar phases such as vermiculitized mica (Fig. 8; Table 3) were only partly misclassified, the rest not finding a match in the internally developed EDS database (Fig. 7E). The abundance of unclassified material (black pixels in Fig. 7E) functioned as an alarm bell. This problem was also encountered by Knappett et al. (2011), who debugged mixed analysis of clay and micrite in calcareous matrices as 'Ca-Al-silicates'. When considering relative abundances of matrix minerals, the comparison between QEMSCAN® and SEM-EDS data revealed that quantitative data provided by the former must be used with caution (Table 4; section 3). Lastly, the automated SEM-EDS mineralogy allowed inferences on homogeneity/heterogeneity of the dark-colored matrix as well as the occurrence of opaque minerals (Fig. 9).

The versatility of the QEMSCAN® tool is further demonstrated by the possibility to reveal shaping and decorating techniques based on void distribution throughout ceramic paste, as clearly highlighted by the analysis of the Petit-Chasseur's pottery. This is powered by the fact that the empty spaces are clearly recognizable in mineralogical maps (Fig. 11). In general, false color mineralogical maps are easily and immediately understood by non-specialists, making the research accessible to a wider scientific community. This includes archaeologists not working in applied geology, as well as to the general public with maps used in the narrative of a museum exposition.

Comparing the kinds of information provided by OM and automated SEM-EDS technology (section 3) it rapidly becomes apparent there are some important discrepancies, which are summarized in Table 5. Various features related to optical properties cannot be observed by means of the phase maps produced by the QEMSCAN® system. These include the color of the matrix and its optical behavior as well as the clasts' internal texture, which allows for inclusion type classification. Similarly, it is not possible to estimate the inclusion quantity by type nor to calculate the grain size distribution of non-plastic components of the ceramic paste. However, automated SEM-EDS technology provides the mineralogy of the clay matrix, automatic modal mineralogy and allows for detecting opaque minerals in dark-colored matrices.

Table 5. Comparison between the kinds of information provided by OM and automated SEM-EDS technology.

	OM	AUTOMATED SEM-EDS TECHNOLOGY
Matrix color	Yes	No
Matrix optical behavior	Yes	No
Matrix homogeneity vs. heterogeneity	Yes	Yes
Matrix mineralogy	No	Yes
Void shape	Yes	Yes
Void size	Yes	Yes
Void orientation	Yes	Yes
Inclusion spatial distribution	Yes	Yes
Inclusion mineralogy	Yes	Yes
Inclusion type/lithology	Yes	No
Inclusion total quantity	Yes	Yes
Inclusion quantity by type/lithology	Yes	No
Inclusion roundness and sphericity	Yes	Yes
Inclusion grain-size distribution	Yes	Yes, but not reliable
Modal mineralogy	No	Yes
Detection of opaque minerals in dark-colored matrix	No	Yes

Finally, the characterization work of the prehistoric pottery from the Petit-Chasseur megalithic necropolis (Carloni et al., 2021) provided relevant data that highlighted the advantages of applying automated SEM-EDS mineralogy in the study of pottery. However, the study exposed that this method is not without limitations (Table 6). The literature (section 2) primarily accentuates the positives of the automated SEM-EDS technology and reported only few disadvantages (Table 1). This work provided a new complete and updated assessment of the pros and cons summarized in Table 6, which outlines the system has more constraints than previously realized. Notwithstanding the several issues related to the use of automated SEM-EDS analysis for the characterization of archaeological ceramics, one should bear in mind that no other technique currently provides such comprehensive information obtained for the Petit-Chasseur ceramics in the time frame of a few hours per sample. The problems encountered when trying to perform a petrographic study of archaeological ceramics by means of the automated SEM-EDS technology are mainly due to the fact that the system does not currently take into account the optical properties of the analyzed material. In conclusion, the use of automated SEM-EDS in archaeometric research of pottery offers a wealth of useful data and will be seen as a standard methodology in future investigation of archaeological material, alongside more traditional techniques such as optical petrography, regular scanning electron microscopy and X-ray diffraction.

Table 6. Updated list of advantages and disadvantages of the use of the automated SEM-EDS technology in the study of archaeological ceramics.

		PREVIOUS WORKS	THIS PAPER
ADVANTAGES	Identical samples may be analyzed by optical microscopy as well as electron microbeam techniques	X	
	Operator-independent analysis, highly reproducible and accurate data	X	
	Quick, automatic and in-depth characterization		X
	Fully quantitative data	X	
	Number of acquired spectra impossible to collect via traditional manual SEM-EDS analysis		X
	Combination of textural and mineralogical data based on elemental spectra	X	
	Mineralogical characterization of finest aplastic inclusions and groundmass	X	
	Possibility to group ceramics according to the mineralogy of their matrix		X
	Study of matrix features hidden by the dark color generated by firing reduced atmosphere	X	
	Characterization of opaque minerals and (semi)-amorphous phases	X	
	Analysis of clay pellets	X	
	Automatically generated modal mineralogy	X	
	Automatically generated modal mineralogy for just one phase or a selection of minerals		X
	Grain-size distribution of a single phase	X	
	Detection of non-stoichiometric firing phases	X	
	Characterization of weathering processes affecting discrete phases and aplastic inclusions	X	
	Compositional grouping	X	
	Identification of phases marking distinct raw material sources	X	
	Comparison of ceramics and hypothesized raw material sources	X	
	Markers of primary and secondary forming techniques	X	
	Different kinds of data obtained all at once	X	
	Mineralogical maps easily and immediately understandable by non-specialists		X
DISADVANTAGES	Misclassification of phases of similar chemistry	X	
	Mineral identification exclusively based on chemical properties, disregarding of the crystallographic and optical ones		X
	Polymorphs unclassified	X	
	Human debug needed when dealing with material of peculiar composition		X
	Debugging procedures require excellent knowledge of phase chemistry		X
	Failure of automated identification of lithoclasts	X	
	Simplification of the lithoclasts' internal texture		X
	Inability to perform a regular grain size distribution of aplastic inclusions		X
	Classification of clay minerals not highly accurate		X
	Miscalculation of relative abundance of the different clay minerals composing the paste		X

5. CONCLUSIONS

The present study demonstrates the usefulness of mineralogical maps, which offer a wealth of information regarding the compositional characteristics of vessels, raw materials used in pottery manufacturing, and technological aspects of ceramic production. The literature review, along with the exemplary case study analyzing the prehistoric pottery from the megalithic necropolis of Petit-Chasseur, allowed for a comprehensive review of strength and limitations of the application of automated SEM-EDS analysis in pottery research.

(i) The automated SEM-EDS solutions are an operator-independent techniques which provide highly reproducible and accurate quantitative data of great statistical significance. Data are quickly acquired and automatically interpreted in a time frame unparalleled by the standard thin-section petrography. However, the data are still to be processed to address the issues of mixed analyses which requires an excellent knowledge of mineral chemistry.

(ii) Acquired EDS spectra are location specific which permits the visualization of various textural features in such acquired phase maps. Mineralogical characterization of opaque minerals and groundmass adds to regular OM-based grouping.

(iii) Phase determination relies on chemical composition only and does not take into consideration crystallographic characteristics, which may lead to misclassification of the phases of similar chemistry (pseudomorphs, clay minerals).

(iv) Lithoclasts' internal texture is highly simplified, hampering the identification of the rock type. Furthermore, the system cannot perform a traditional grain size distribution of aplastic inclusions and morphometric studies can be executed on individual crystals only.

(v) Due to extremely rapid EDS spectra acquisition the classification of the clay minerals may not be comparable to the one performed by the conventional SEM-EDS and therefore the relative abundance of sheet silicates calculated by the system may not be reliable.

(vi) Modal mineralogy for each ceramic thin section is automatically computed and can serve as a base for compositional grouping.

(vii) The automated SEM-EDS technology provides a complete advanced mineralogical characterization, and a variety of information that permits an inquiry into raw material selection, procurement, and use as well as pots' shaping technique.

(viii) The use of automated SEM-EDS in archaeometric research of pottery nevertheless offers a wealth of useful data and should be incorporated as a standard method in the future investigation of archaeological material, alongside the more traditional techniques such as optical petrography, regular scanning electron microscopy and X-ray diffraction.

AUTHOR CONTRIBUTIONS

Conceptualization, Delia Carloni; methodology, Delia Carloni, Branimir Šegvić, and Giovanni Zanoni; software, Delia Carloni, Branimir Šegvić, and Giovanni Zanoni; validation, Mario Sartori and Marie Besse; formal analysis, Delia Carloni, Branimir Šegvić, and Giovanni Zanoni; investigation, Delia Carloni, Branimir Šegvić, and Giovanni Zanoni; resources, Marie Besse; data curation, Delia Carloni, Branimir Šegvić, and Giovanni Zanoni; writing—original draft preparation, Delia Carloni; writing—review and editing, Branimir Šegvić, Giovanni Zanoni, Mario Sartori, and Marie Besse; visualization, Delia Carloni; supervision, Branimir Šegvić, Giovanni Zanoni, Mario Sartori, and Marie Besse; project administration, Marie Besse; funding acquisition, Marie Besse. All authors have read and agreed to the published version of the manuscript.

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