

Céramiques imprimées de Méditerranée occidentale (VI^e millénaire AEC) : données, approches et enjeux nouveaux / Western Mediterranean Impressed Wares (6th millennium BCE):

New data, approaches and challenges

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From geomaterials to archaeological pastes: the example of “granitic” pottery productions from Neolithic Castellar - Pendimoun (Alpes-Maritimes, France)

Jean-Marc LARDEAUX, Gilles DURRENMATH, Marzia GABRIELE,
Chrystèle VERATI and Didier BINDER

Résumé : Le site de Castellar – Pendimoun offre des jeux de données qui font référence dans le contexte de la mise en place des premières communautés agropastorales néolithiques dans le domaine liguro-provençal. Les assemblages céramiques provenant des horizons de l'*Impresso-Cardial* sont caractérisés par trois types de pâtes, glauconieuses, granitiques et mixtes. Notre étude porte sur les pâtes granitiques dont l’abondance relative caractérise les horizons les plus anciens (*Impressa*, phases PND-1A et 1B) et dont les sources potentielles se trouvent à plusieurs dizaines de kilomètres du site. Nos travaux combinent observations géologiques naturelles (minéralogie, pétrographie et microstructures de déformation des minéraux et des roches), analyses géochimiques quantitatives (ICP-AES et ICP-MS pour les éléments majeurs et les éléments en traces, géochronologie ^{40}Ar - ^{39}Ar), production expérimentale, à différentes températures (300°C, 500°C et 700°C), de céramiques à partir des matières premières considérées comme les plus plausibles dans le cas d’étude et modélisations thermodynamiques quantitatives de la nature des assemblages minéralogiques les plus stables en fonction des compositions chimiques spécifiques des matières premières considérées et des variations de la température. L’étude, au microscope polarisant, de dix-sept lames minces des céramiques archéologiques révèle une importante quantité, *ca.* 40 %, de particules non plastiques. Les observations minéralogiques et pétrographiques démontrent que les minéraux et les lithoclastes présents dans ces céramiques archéologiques sont caractéristiques des granites subsolvus à deux micas. Au sein de ces céramiques, les quartz montrent des textures de déformation ductile intra-cristalline (extinctions roulantes, formation de sous-grains plus ou moins fortement désorientés, recristallisation dynamique de nouveaux grains avec forte réduction de taille), les micas montrent des extinctions roulantes et des *kink-bands* alors que les feldspaths montrent des macles de déformation, des extinctions roulantes et de nombreuses micro-fractures scellées par des micas. Ces microstructures sont caractéristiques de la déformation des roches granitiques dans des conditions de température comprises entre 350 et 450°C, c’est à dire dans le faciès des schistes verts. Dans de telles conditions la déformation des granites est localisée dans des couloirs mylonitiques au sein desquels les roches montrent une forte réduction de la taille des grains de quartz et le remplacement des feldspaths par des micas blancs. Ces types de microstructures sont observés dans les minéraux et les lithoclastes des céramiques étudiées démontrant que les terres d’altération utilisées pour la fabrication des poteries dérivent de granites leucocrates à deux micas déformés (mylonites) dans le faciès des schistes verts. Les céramiques contiennent de plus de rares fragments de granophyres, de lamprophyres et de migmatites à sillimanite. Dans le contexte géologique régional quatre domaines montrent des roches granitiques et métamorphiques à l’affleurement : les massifs de Calizzano et Savona en Ligurie, les formations détritiques éocènes et oligocènes de la série des grès d’Annot, le massif des Maures-Tanneron et enfin le massif de l’Argentera-Mercantour. Une analyse comparative détaillée des caractéristiques géologiques de ces différents domaines désigne le granite central de l’Argentera-Mercantour (avec ses couloirs de mylonites, ses filons de lamprophyres et ses enclaves de migmatites) comme la géo-ressource la plus plausible utilisée pour la fabrication des céramiques de Pendimoun. Les analyses géochimiques et géochronologiques à haute résolution effectuées d’une part sur les terres d’altération dérivant des mylonites du granite central de l’Argentera et d’autre part sur les céramiques archéologiques montrent de remarquables similitudes. Ces terres d’altération ont donc été utilisées afin de produire des céramiques expérimentales avec des cuissages à différentes températures. La minéralogie de ces céramiques évolue de façon significative avec la température. Dès 500°C les argiles ont été en totalité transformées en micas, les chlorites sont partiellement recristallisées en biotites et les néo-cristallisations de quartz, muscovite, biotites, ilménite sont nombreuses dans la matrice comme aux interfaces entre les phases magmatiques initiales. Les évolutions minéralogiques observées, entre 300 et 700°C, dans les céramiques expérimentales sont cohérentes avec les données pétrologiques issues des études du métamorphisme de contact de roches sédimentaires argileuses. Cependant comme les conditions de stabilité des minéraux sont fortement dépendantes des compositions chimiques spécifiques

des méta-sédiments nous avons réalisé une modélisation thermodynamique de stabilité des assemblages minéralogiques, sur une large gamme de températures, sur deux exemples de terres d'altération dérivant du granite central de l'Argentera et dont les compositions chimiques ont été analysées. Les résultats de la modélisation thermodynamique sont en bon accord avec les observations effectuées sur les céramiques expérimentales et permettent de fixer les conditions de cuisson les plus probables des céramiques archéologiques entre 550 et 500°C. Cette approche multi-méthodes nous permet de mettre en évidence la diversité des transformations minéralogiques se déroulant, au sein des terres d'altération, d'une part lors du cycle géologique « naturel » (exhumation, déformation et altération des roches) et d'autre part lors du « cycle industriel » (cuisson en fours à différentes températures). Ces résultats ouvrent de nouvelles perspectives sur la méthodologie à mettre en œuvre afin de caractériser, de façon rigoureuse, la nature des matières premières ayant servi à la fabrication des céramiques archéologiques. Nous démontrons que les matrices, brunâtre à rouge foncé, des céramiques de Pendimoun sont constituées de minéraux néoformés, en particulier des biotites, ayant cristallisé durant le processus de cuisson. La disparition de toutes les argiles et d'une grande partie des chlorites dès 500°C peut conduire à une sous-estimation de la proportion des phases argileuses initialement présentes dans la géo-ressource. Les méthodes de mise en forme des poteries du Néolithique pourraient être examinées à la lumière de ces résultats. Enfin, nos travaux soutiennent l'hypothèse de la collecte de sols à une altitude relativement élevée (*ca.* 2000 m) modifiant la vision d'un peuplement néolithique précoce confiné à la seule frange côtière.

Mots-clés : géo-ressources, minéralogie, géochimie quantitative, modélisation thermodynamique, pâtes granitiques, mylonites, sites de haute altitude.

Abstract: This study provides new insights into the “granitic” pottery pastes from the Neolithic Castellar – Pendimoun site by combining natural geological observations (mineralogy, petrography, microstructures), quantitative geochemical investigations, experimental firing of putative raw material at different temperatures to produce experimental pottery and quantitative thermodynamic modelling of the most stable mineral assemblages, within a large temperature range, for the specific chemical composition of the alteration soils used for ceramic production. First, it could be stated that the main geological source for raw materials used to produce the Pendimoun granitic pastes was leucocratic two-micas granite which was deformed, within mylonitic shear zones, under greenschist facies conditions. It could be demonstrated that the most possibly used resource within the geological environment was the deformed « Argentera Central Granite », with its associated dykes and surrounding migmatites. It was also possible to highlight the chemical compatibility of the early potteries from Pendimoun with the Argentera-Mercantour altered granite deposits and these raw materials were used to produce experimental ceramics. The experimentally produced mineral assemblages are consistent with the petrologic constraints imposed by the study of naturally metamorphosed clay-rich sedimentary rocks. Thermodynamic modelling performed on two alterite samples yielded results that are consistent with our experiments and show that the potteries found at Pendimoun were produced at a temperature close to 550–500°C. This dataset makes it possible to decipher the mineral reactions that may occur during the “natural cycle” (exhumation, deformation and alteration of the rocks) and the “firing cycle” and opens up new perspectives for geological sourcing and analysis as regards the methods of shaping and firing Neolithic pottery. Finally, our work supports the hypothesis of soils collection at a relatively high altitude (*c.* 2000m) modifying the view of an early Neolithic settlement confined to the coastal fringe.

Keywords: geological sources, mineralogy, quantitative geochemistry, thermodynamic modelling, granitic pastes, mylonites, high-altitude sites.

INTRODUCTION

During the first half of the 6th millennium BCE, a particular mode of pottery production illustrates the specificity of the pioneer Neolithic diffusion pattern across the Tyrrhenian Sea, towards the North-Western Mediterranean, and reveals cultural traditions that are distinct from those observed eastwards, *i.e.* in the Aegean-Balkan and Adriatic areas, which are thought to be at the origin of the Western Impressed Wares (Gomart *et al.*, 2017, Gomart, Binder, Gabriele *et al.*, this volume). This singular method of pottery making strongly contrasting with the huge diversity of shapes and decorations modes observed in this area highlights the diversification of the pottery assemblages over time and space (Manen *et al.*, 2019 and this volume).

In the Tyrrhenian area, the wide range of raw materials selected for the shaping of the earliest Neolithic vessels equally emphasises such a diversity (*e.g.* Binder, 1991; Échallier and Courtin, 1994; Manen *et al.*, 2006; Capelli *et al.*, 2007; Gabriele, 2014 and this volume). In general, the frequent use of mineral rich, coarse or very

coarse pastes, contrasts with the Aegean-Balkan tradition, with potters commonly favouring plastic carbonate clays (Spataro, 2002; Muntoni, 2003; Spataro, 2017). The impressive raw material diversity, observed for instance in the northern Tyrrhenian Sea and the gulf of Lion, makes it possible to assume that the choice of raw materials for pottery manufacturing was not only dictated by local availability but also reflects more complex processes, involving the functional or symbolic sphere. Indeed, it raises questions with regard to the differentiated use of materials to obtain a certain type of shape and /or physical properties for a specific function (*e.g.* Binder, 1991) and with regard to the transfer of raw materials, pastes or pots even over long or very long distances (Capelli *et al.*, 2006 and 2007; Convertini and Bruxelles, 2010; Capelli *et al.*, 2011; Gabriele *et al.*, 2019 and this volume).

Amongst the large array of raw materials available in eastern Provence and Liguria, the presence of pastes containing micaceous minerals stemming from the weathering of materials of granitic or metamorphic origin was noted very early on. As a matter of fact, in his pioneer study of the Cardial series of the Saint-Vallier-de-Thiey – Lombard cave, J.-C. Échallier stressed the paradoxical

aspect of the thin sections of pots made from micaceous *terrae*: whatever the magnification, the matrix appeared as a felting of minerals, *i.e.* small-sized and xenomorphic biotites, suggesting an alteration without significant displacement from the bed rock (Échallier in Binder, 1991). The very low frequency of clayey minerals raised pivotal questions, asked during the drafting of the CIMO project, about the workability of such kind of pastes and subsequently the specific methods that may have been implemented for shaping and firing the pots.

The Pendimoun rock-shelter holds a key position as regards the communication routes between Liguria and eastern Provence and alongside the Finale Ligure – Arene Candide cave site it is a major reference for establishing the framework of the process of neolithisation in the North-Western Mediterranean area (Binder *et al.*, 1993; Maggi and Starnini, 1997; Binder and Maggi, 2001; Panelli, 2019; Binder *et al.*, 2020; Maggi *et al.*, 2020). Thus, both pottery series and associated raw materials have been highly investigated (Basso *et al.*, 2006; Capelli *et al.*, 2007 and 2011; Gabriele, 2014; Pradeau *et al.*, 2016; Gabriele *et al.*, 2020 and this volume).

At Castellar – Pendimoun, these investigations made it possible to identify three groups of pastes used for the manufacturing of the Neolithic potteries: glauconitic, granitic and mixed pastes (*i.e.* glauconitic-granitic pastes with deliberate mixing; Gabriele, 2014). Detailed petrographic, mineralogical and geochemical analyses of the pottery clearly established that local glauconite-bearing marls were used for glauconitic pastes production (Basso *et al.*, 2006; Gabriele, 2014; Gabriele *et al.*, this volume) and that this glauconitic material was also used for its colouring properties (Pradeau *et al.*, 2016). Moreover, petrographic analysis using an optical microscope and a SEM demonstrate that the mixed pastes resulted from anthropogenic production (Gabriele, 2014; Gabriele *et al.*, this volume).

The “granitic” pottery pastes throw up challenging issues concerning both their intrinsic properties and the range of their transfer. Based on the well documented case study offered by the Pendimoun series, the aim is therefore to present detailed mineralogical, petrographic, microstructural and geochemical analyses of granitic pastes to determine their putative geomaterial sources. Beyond the identification of the potential outcrops and the consequences in terms of mobility and trade, the aim of this paper is also to document the effects of both the natural geological cycle and the firing cycle on the mineralogical evolution of raw materials and thus on the characterisation of archaeological pottery.

ARCHAEOLOGICAL CONTEXT

The site of Castellar – Pendimoun (Alpes-Maritimes) is located at the foot cliff of the Roc de l’Ormea massif at c. 700m asl, c. 4km north of Menton and less than 1km from the French-Italian border (fig. 1). From this rock

shelter, which opens to the west, it is possible to observe the Mediterranean Sea to the south and the mountains that run parallel to the Careï River to the west, an axis of natural penetration towards the north and the alpine reliefs, through the alpine Colle di Tenda pass towards the Po basin.

The rock-shelter opened within Upper Jurassic limestones alternating with marly limestones, marls and schistose marls including glauconite-bearing levels attributed to the Lower Cretaceous (Gèze *et al.*, 1968). These formations constitute the roof and the base of the rock shelter respectively. This overhang is at the origin of an important circulation of water and springs formation at the junction, and of the development of large spathic calcite prisms (5-10cm long), which are close to the optical quality and are also of some interest for pottery production within diverse cultural contexts. Overlooking the site, at less than 1km away, there are the extended formations of the undifferentiated Upper Cretaceous limestone with glauconitic horizons.

Pendimoun displays a set of Holocene stratified levels and structures (hearts, pits, postholes, burials, etc.), overlaying thick gravels deposits. Within the latter, possible Tardiglacial and/or Pleistocene occupational layers have not yet been explored.

Subsequent to a poorly preserved occupation of the Early Mesolithic (phase 0, 8240-7590 BCE) followed by a long gap, the core of the sequence is represented by the Impressed Ware Culture. The latter is divided in two main successive aspects: *Impressa* (phase 1A, 5720-5660 BCE; phase 1B, 5650-5440 BCE) and Cardial (phase 2A, 5440-5200 BCE; 2B, 5330-5020 BCE). At the end of the 6th millennium BCE the site reveals influences from the Po plain (*i.e.* Vhò group) and transitional aspects towards the Square Mouthed Pottery Culture (phase 3, 5150-4680 BCE). The top of the sequence provides traces of occupation related to successive aspects of the Southern Chassey culture (phase 4) and to the Bell Beaker (phase 5), and sporadic finds dated to the Bronze Age, the Roman Era, as well as to the Middle Ages and the Modern period (Binder *et al.*, 1993; Binder and Lepère, 2014; Binder *et al.*, 2020, Binder, Gomart *et al.*, this volume).

The large quantity of pottery, both sherds and individual pots, and the consistency of the successive assemblages apparently made from a very limited number of raw material, have made the *Impressa* and Cardial pottery series a challenging case study for understanding the relations between natural resource, shaping methods, shapes, decoration and uses (Gabriele, 2014; Gomart *et al.*, 2017; Gomart, Binder, Gabriele *et al.*, this volume; Drieu *et al.*, 2019, 2021, and this volume; Cassard, 2020 and this volume).

MINERALOGIC-PETROGRAPHIC CHARACTERISATION OF THE NEOLITHIC POTTERY OF PENDIMOUN

The *Impresso*-Cardial levels from Pendimoun yielded large series of sherds (several thousands) which were

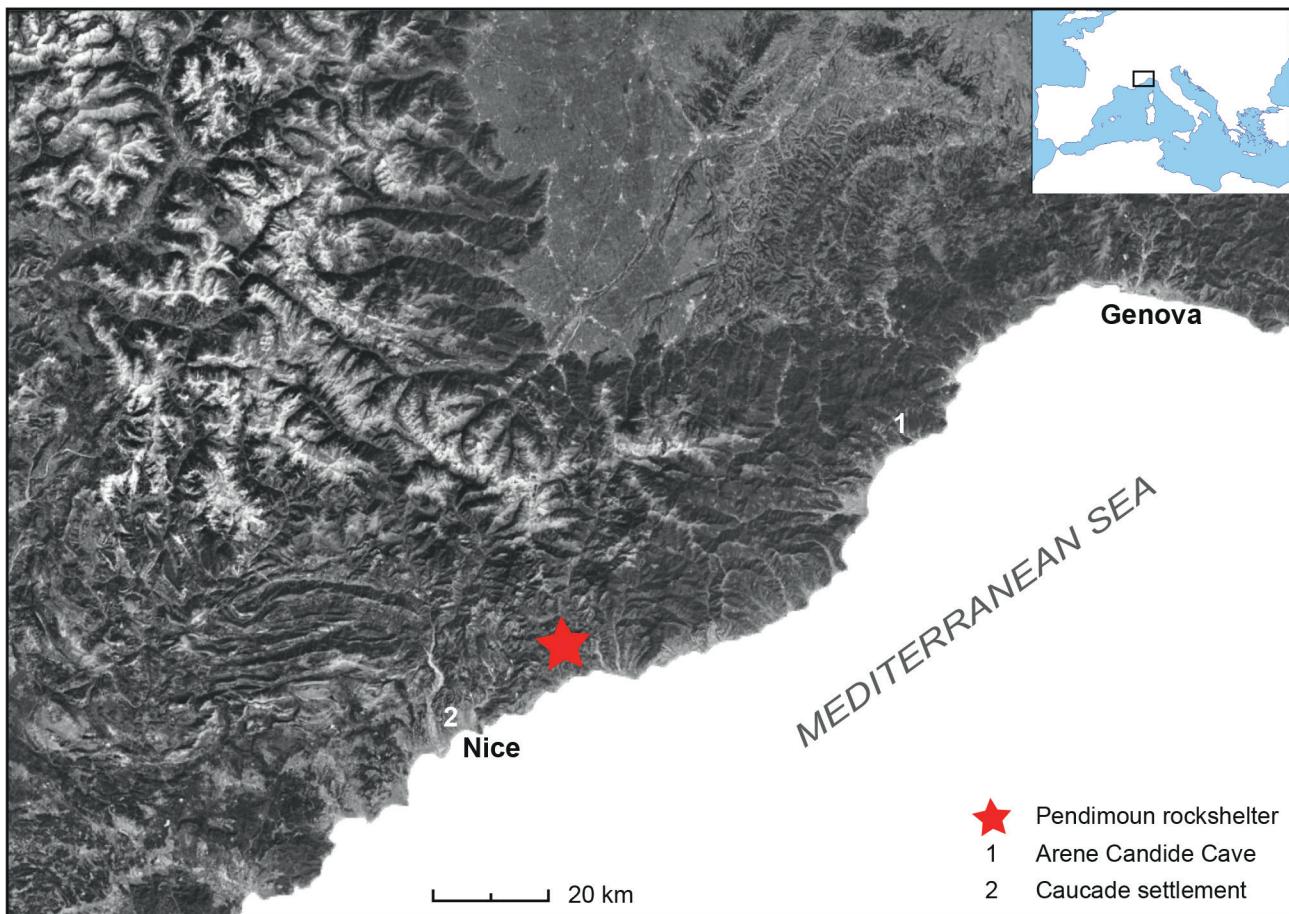


Fig. 1 – Location of the Castellar - Pendimoun rock shelter (red star) and the related *Impressa* Neolithic sites of Finale Ligure - Arene Candide cave (1) and Nice - Caucade (2).

Fig. 1 – Localisation de Castellar - abri Pendimoun (étoile rouge) et des sites du Néolithique *Impressa* de Finale Ligure - Arene Candide cave (1) et de Nice – Caucade (2).

systematically observed with a stereoscope at a low magnification (<50) and more than two hundred pots ($n = 208$) were identified within the *Impressa* phases PND-1A, PND-1B and PND-1-individued ($n = 125$) and within the Cardial phases PND-2A and PND-2B ($n = 83$; Gabriele, 2014; Gabriele *et al.*, this volume). Among these specimens seventeen ($n = 17$) samples were selected for petro-mineralogical analyses from which twenty-five thin sections ($n = 25$) were made: twenty-two ($n = 22$) radial thin sections, covered, and three ($n = 3$) tangential ones, uncovered. Thin section analyses of these archaeological specimens confirm the three main groups of archaeological pastes observed at a mesoscopic scale (granitic, glauconitic and ‘mixt’, fig. 2) and an additional single pot made from pastes including specific Triassic minerals (Gabriele *et al.*, this volume).

Observation of thin sections under a polarised microscope (fig. 3AB) reveals that the granitic pastes contain a significant amount, *i.e.* 40% (Quinn, 2013), of non-plastic particles. The latter include both isolated minerals and rock fragments (*i.e.* lithoclasts) with single-spaced distribution type (unimodal distribution).

The isolated phases exhibit an irregular, angular, and less frequently subrounded morphology (fig. 3BD).

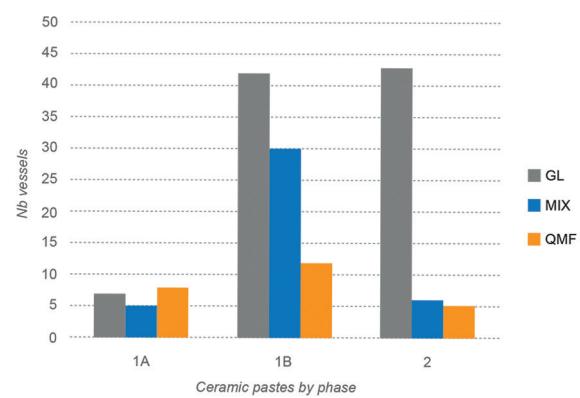


Fig. 2 – Castellar - Pendimoun, distribution of the vessels according to the main types of pastes (GL: “glauconitic”; QMF: “granitic”; MIX: “mixt”) and to the chronocultural phases (1A and 1B: *Impressa* phases; 2: Cardial phase).

Fig. 2 – Castellar - Pendimoun, distribution des récipients en fonction des principaux types de pâtes (GL : « glauconieuse » ; QMF : « granitique » ; MIX : « mixte ») et des phases chronoculturelles (1A et 1B : *Impressa* ; 2 : Cardial).

They range in size from a dozen microns to one millimetre and consist in order of decreasing abundance of quartz, K-feldspar (sometimes with perthites),

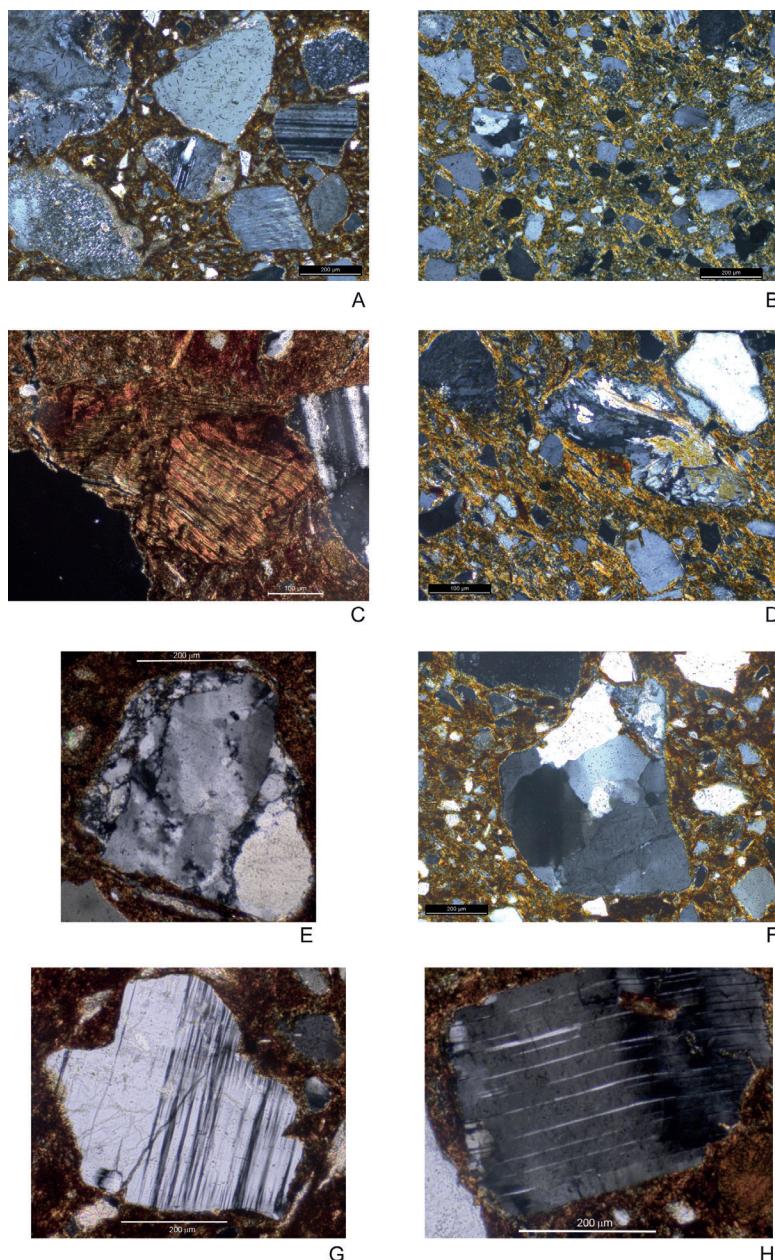


Fig. 3 – Castellar - Pendimoun, mineral assemblages and microstructures characterizing the “granitic” pastes. All pictures are taken by plane polarised light. A: large-sized isolated grains of quartz, plagioclase and K-feldspar in brown, biotite-rich, matrix; note the development of thin coronas of micas and quartz at the rims these non-plastic particles. B: isolated grains of quartz and feldspar with contrasted sizes and morphology within light-brown matrix. C: isolated grains of biotite and plagioclase in reddish, biotite-rich, matrix; note the intra-crystalline deformation (undulose extinction) of the biotite grain. D: lithoclast composed of recrystallized aggregate of quartz, K-feldspar and plagioclase with fibrous sillimanite (in yellow to white interference colors) and isolated quartz and feldspar grains in a biotite-rich matrix. E: typical microstructure of quartz ductile deformation with a grain showing subgrains (with contrasted greys interference colors) in its core and small-sized recrystallized new grains at crystal rims. F: intra-crystalline deformation in a quartz grain with significant misorientation of subgrains and thus sharp subgrains boundaries. G: deformation twins in plagioclase. H: undulose extinction in a plagioclase grain; note small-sized micro-cracks sealed by micas and quartz.

Fig. 3 – Castellar - Pendimoun, assemblages minéralogiques et microstructures caractéristiques des pâtes « granitiques » (lumière polarisée analysée). A : quartz, plagioclase et feldspath potassique, en grains isolés et de grande taille, au sein d'une matrice brune riche en biotite ; noter le développement de fines couronnes de réaction, avec micas et quartz, aux limites de ces minéraux. B : grains isolés de quartz et de feldspath montrant une diversité de tailles et de formes au sein d'une matrice brun clair. C : biotite et plagioclase en grains isolés au sein d'une matrice rouge foncé riche en biotite ; noter la déformation intra-cristalline (extinction roulante) de la biotite. D : lithoclaste composé de quartz recristallisé, de feldspath potassique et de plagioclase avec des fibres de sillimanite (teintes de polarisation jaune à blanc) et associé à des grains isolés de quartz et feldspath au sein d'une matrice riche en biotite. E : microstructure typique de la déformation ductile du quartz avec formation de sous-grains (avec des teintes de polarisation variables dans les gris du premier ordre) au cœur du cristal et de petits grains recristallisés (néo-grains) en bordure du cristal. F: déformation intra-cristalline d'un grain de quartz montrant des sous-grains avec une forte désorientation et donc des limites nettes. G : macles de déformation dans un plagioclase. H : grain de plagioclase montrant une extinction roulante ; noter la présence de micro-fractures scellées par des micas et du quartz.

plagioclase, muscovite, and biotite. However, in some samples, muscovite is more abundant than the two feldspars. Occasionally, rare tourmaline, zircon, titanite and one garnet grain were observed. All major phases show textural evidence of intra-crystalline deformation, well known in naturally high-strained rocks (see Passchier and Trouw, 2005 with references therein). Quartz grains display marked undulose extinctions, intracrystalline subgrains with frequent rotations of subgrain boundaries and evidence for dynamic recrystallisation with the development of aggregates of small-sized new grains (fig. 3EF). K-feldspars, which are mainly orthoclase and less frequently microcline, deformed under a brittle-ductile regime evidenced by undulose extinctions and intragranular micro-fracturing. The fractures can be sealed by quartz or white micas and orthoclase grains are frequently partly altered and replaced by aggregates of sericite and clays minerals. Plagioclase exhibit undulose extinctions, deformation twins (fig. 3GH) and some recrystallisations into small-sized white micas. Muscovites are often elongated, up to 800 microns in size, with kinking leading development of undulose extinctions. Biotite grains, partially oxidised, display undulose extinctions (fig. 3C) and are occasionally transformed into chlorite fibres. Occasionally, large-sized feldspar or quartz grains are rimmed by thin aggregates of micas and quartz (fig. 3A)

The lithoclasts display irregular shapes and vary in size from c. 300 microns to a few millimetres. They mainly consist of fragments of two-micas granites, within which quartz is the most abundant phase, followed by K-feldspar and plagioclase and minor muscovite, biotite and zircon. In these granitic fragments, all the major phases display textures of intra-crystalline deformation similar to those described above for isolated grain minerals. It was also possible to identify fragments of granitic composition, with rare biotite and exceptionally garnet, but with fine-grained textures typical of aplitic dykes. In addition to these two types of lithoclasts a single fragment of subvolcanic granitic rock with typical granophytic texture, and one fragment of a melanocratic mafic magmatic rock were observed. In this latter, sub-millimetric phenocrysts of brown amphibole, plagioclase, oxides and clinopyroxene occur in a microcrystalline groundmass rich in oxides (ilmenite, magnetite), small laths of plagioclase and rare oxidised biotite diagnostic for lamprophyre dykes. Furthermore, to be exhaustive it is necessary to emphasise the occurrence of one lithoclast of quartzo-feldspathic leucosome with typical granoblastic texture and composed of recrystallised aggregate of quartz, K-feldspar and plagioclase with fibrous sillimanite (fig. 3D), typically described in migmatites.

The matrix corresponds to the reddish to light-brown typology (Échallier and Courtin, 1994) and consists in fine-grained biotites with minor and small-sized ilmenite, quartz and feldspar grains (fig. 3B, D and F). Occasionally, interstitial calcite was observed.

Following this description, it is interesting to note that the non-plastic minerals and lithoclasts identified in the “granitic” fraction of the mixed pastes are similar,

in terms of both their nature and their textures, to those described above with the exception of fragments of sub-volcanic mafic rock and sillimanite-bearing leucosome, unknown in these mixed pastes.

DETERMINATION OF POSSIBLE RAW MATERIALS USED FOR THE GRANITIC PASTES OF PENDIMOUN

The geological constraints to take into account for sourcing

If we take into account all the petrographic and mineralogical data discussed in the previous paragraph, the main geological source for raw materials used to produce the Pendimoun granitic pastes is clearly, from a lithological point of view, a leucocratic subsolvus two-micas granite, in which muscovite is more abundant than biotite, and in spatial association first with aplites, sometimes garnet-bearing, and lamprophyres dykes, and second with sillimanite-bearing migmatites (enclaves or surrounding rocks?).

Moreover, this material must have undergone ductile deformation under specific conditions. Indeed, the coexistence within the same rock sample of textures showing sub-grains formation and dynamic recrystallisation of quartz, undulose extinctions and intragranular micro-fractures in K-feldspar, deformation twins in plagioclase and finally kink-bands and undulose extinctions in micas (fig. 3C and E-H) is diagnostic for ductile deformation under a temperature range between 350 and 450°C (Hobbs *et al.*, 1976; Poirier, 1985; Urai *et al.*, 1986; Bell and Johnson, 1989; Tullis, 1990; Stipp *et al.*, 2002; Passchier and Trouw, 2005; Hentschel *et al.*, 2019). In other words, the main geological source is a leucocratic two-micas granite deformed under greenschist facies conditions. It has long been established that under greenschist facies conditions solid-state deformation of granites results in heterogeneous finite strain patterns, high strains being localised within networks of shear zones (Mitra, 1978 and 1979; Ramsay and Alison, 1979; Choukroune and Gapais, 1983; Simpson, 1985; Cobbold and Gapais, 1987; Gapais, 1989). Within shear zones the most deformed rocks exhibit a mylonitic texture characterised by significant grain-size reduction driven either by dynamic recrystallisation of softer phases or by reaction softening (*i.e.* destabilisation and replacement) of harder phases (Teall, 1885; Spry, 1969; Ramsay and Graham, 1970; Ramsay, 1980). This applies to the Pendimoun granitic pastes with aggregates of small-sized dynamically recrystallised quartz grains, partial replacement of feldspars by white mica (*i.e.* sericite) and concentration of stretched and plastically deformed fibres of muscovite.

The putative sources in the available regional geological framework

In the geological framework of interest, four candidates can be considered as putative sources of raw

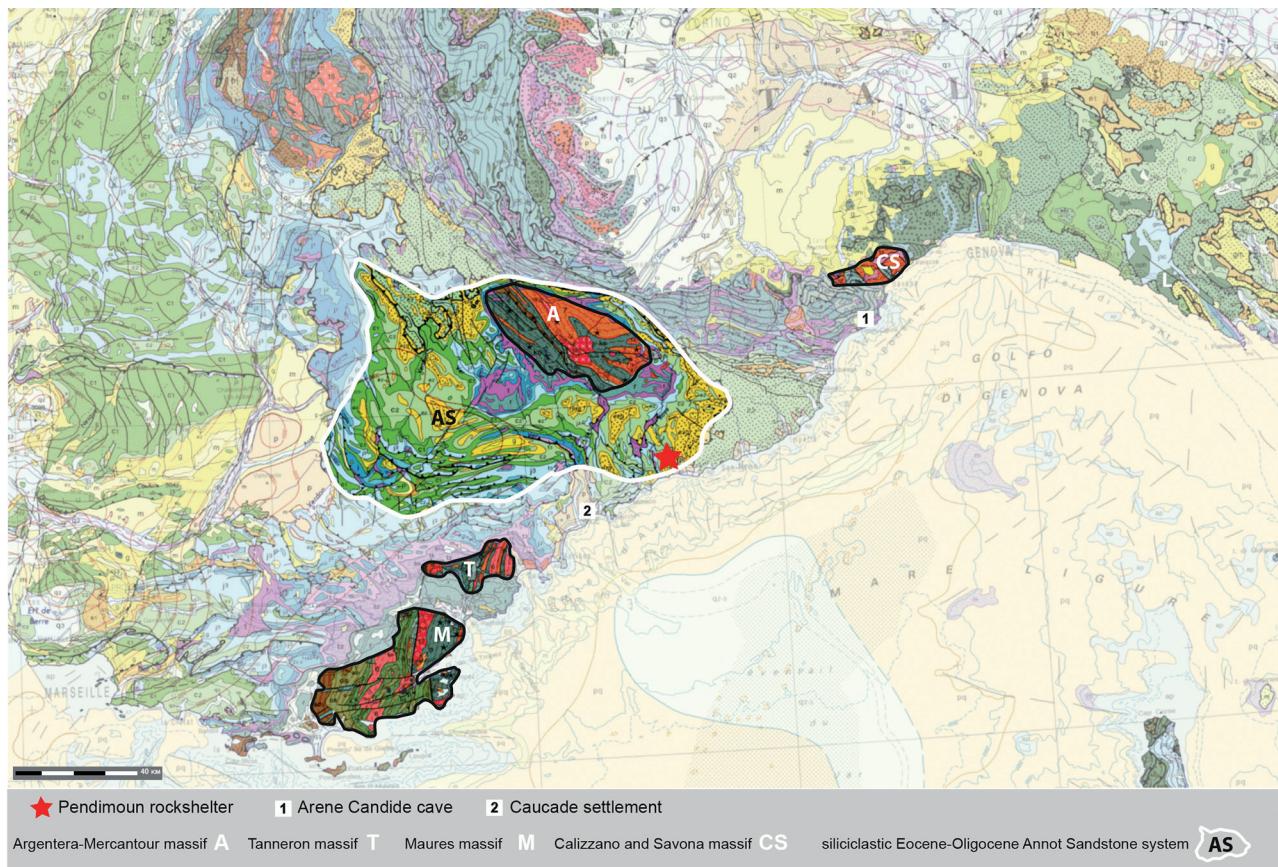


Fig. 4 – Main potential sources for the pottery production of Pendimoun located on the BRGM 1:100,000 geological map of France (Chantraine et al., 1996. For the interpretation of the colour references in this figure, the reader may refer to the web version of this article.)

Fig. 4 – Localisation des principales sources potentiellement utilisées pour les productions de poterie de Pendimoun sur la carte géologique au 1/100 000 de la France (Chantraine et al., 1996. Pour l'interprétation des couleurs de cette carte le lecteur peut se référer à la version électronique de ce document).

material sources used for production of granitic ceramics at Pendimoun (fig. 4).

In the Ligurian Alps, the Calizzano (60km away from the site) and the Savona (90km away) crystalline massifs represent two pieces of the orogenic Variscan crust, including granites and migmatites, severely reworked during Alpine orogeny and considered as “allochthon basement units” of the Briançonnais zone in Liguria (Haccard et al., 1972; Cortesogno et al., 1982; Vanossi et al., 1980 and 1984; Vanossi and Goso, 1983; Capponi and Crispini, 2002). These crystalline basement units are quite often associated with levels of Permian rhyolites and andesites and with sediments typical of the so-called Briançonnais sedimentary series, also strongly deformed and metamorphosed during Alpine orogeny and thus transformed into meta-rhyolites, meta-andesites and meta-sediments. Given that they are located within the internal domain of the Alpine belt, all these lithologies were metamorphosed under high-pressure greenschist facies and/or low-temperature blueschist facies (Cortesogno et al., 1982; Goffé, 1984; Messiga, 1987; Bigi et al., 1990; Goffé et al., 2004; Lardeaux, 2014) and display – depending on the lithology considered – diagnostic metamorphic phases such as carpholite, pyrophyllite, phengite, chloritoïde, zoïsite or clinzoïsite, glaucophane and/or crossite

or barroisite, garnet for example. The Variscan granites were mainly transformed in orthogneisses, and well-preserved granitic textures are rare, while the migmatites are clearly poly-metamorphic (Del Moro et al., 1981). The lack of alpine high-pressure metamorphic phases as well as of fragments of meta-rhyolites, meta-andesites or HP/LT meta-sediments in the pastes of Pendimoun makes the hypothesis of a Ligurian provenance unlikely.

In eastern Provence three geological candidates display granitic material: the siliciclastic Eocene-Oligocene Annot sandstone system, the Argentera-Mercantour massif and the Maures-Tanneron massif (Kerckhove et al., 1979; Rouire et al., 1980).

The Annot sandstone system is the last member of the well-known Nummulitic Trilogy deposited within the foreland basins of the Alpine belt during progressive alpine continental collision (Faure-Muret et al., 1956; Bouma, 1962; Ford et al., 1999; Joseph and Lomas, 2004). In the area of study, these sand-rich turbidite sediments outcrop in synclines within a 5 to 20km range around the Pendimoun rock shelter (Lanteaume, 1962; Gèze et al., 1968; Campredon, 1972; Rouire et al., 1980). The source of the sediment supply to this turbidite system is attributed to Variscan and Permian Corsica-Sardinian massifs (Ivaldi, 1974; Campredon, 1972; Sinclair, 1997;

Campredon and Giannerini, 1982; Jean, 1985; Garcia *et al.*, 2004). So far, not a single fragment of greenschist facies mylonitic granite has yet been described in the Annot sandstone system. Moreover, the Corsica-Sardinian massifs display specific magmatic rock associations, particularly two-micas granites, granodiorites, leucogranites, Mg-K rich granites, diorites, migmatites but also a lot of syenites, alkaline rhyolites, ignimbrites, alkaline basalts and riebeckite, hastingsite and/or aegyrine bearing-alkaline granites (Bonin and Vellutini, 1976; Orsini, 1980; Rossi and Rouire, 1986; Cappelli *et al.*, 1992; Carmignani *et al.*, 1994 and 2001; Cocherie *et al.*, 1994 and 2005; Lardeaux *et al.*, 1994; Bonin *et al.*, 1998; Carosi *et al.*, 2009; Rossi *et al.*, 2009; Casini *et al.*, 2012). The lack of Mg-K rich granites, diorites, syenite, alkaline rhyolite, ignimbrites and alkaline hypersolvus granite in the pastes of Pendimoun as well as the absence of mylonitic granite in the siliciclastic Eocene-Oligocene turbidites prevent these folded sedimentary basins from being considered as putative procurement areas.

The Maures-Tanneron massif (also known as “Crystalline Provence” and located 50 to 100km away from the Pendimoun site) consists of Pre-Permian low-grade to high-grade Variscan metamorphic rocks intruded by granites and overlie by coal basins of Pennsylvanian age (Demay, 1926a and 1926b; Bordet, 1957; Seyler, 1986; Crévola and Pupin, 1994; Toutin-Morin *et al.*, 1994). The orogenic evolution was completed by Permian volcano-sedimentary deposits (Rouire *et al.*, 1980; Crévola *et al.*, 1991). It has now been well established that the Maures-Tanneron massif formed a unique domain with Sardinia and Corsica at Variscan times (e.g. Edel *et al.*, 1981, 2014 and 2018; Elter *et al.*, 2004; Bellot, 2005; Corsini and Rolland, 2009). The global tectonic architecture of the Maures-Tanneron massif displays an asymmetric fan shape involving a western external zone and eastern internal zone separated by the La-Garde-Freinet/Cavalaire thrust zone (Corsini *et al.*, 2010; Schneider *et al.*, 2014). The external zone is formed by a folded pile of units of low-grade metamorphism in the outermost part evolving towards medium to high-grade metamorphism when approaching the internal zone (Gueirard *et al.*, 1970; Bronner *et al.*, 1971; Bordet *et al.*, 1976; Crévola and Pupin, 1994; Seyler and Crévola, 1982; Bellot *et al.*, 2000 and 2002; Corsini and Rolland, 2009; Rolland *et al.*, 2009). The internal zone consists of sillimanite-rich migmatites, including relicts of retrogressed eclogites and serpentinised peridotites, intruded by late-orogenic calcalkaline and peraluminous granites, cordierite-bearing leucogranites and tonalites (see reviews in Schneider *et al.*, 2014 and Gerbault *et al.*, 2018, with references therein). This internal domain is crosscut by a major ductile strike-slip shear zone, the Joyeuse–Grimaud fault, in which the syn-tectonic Rouet-Plan de la Tour pluton is emplaced (Onezime *et al.*, 1999; Corsini and Rolland, 2009; Corsini *et al.*, 2010). This latter consists of a zoned pluton composed of a biotite-rich peraluminous, sometimes cordierite-bearing, granite intruded by dykes of leucogranites, aplites and pegmatites. It is also closely associated with tonalites intrusions. Furthermore,

this pluton exhibits K-feldspars rich cumulative facies and numerous fine-grained diorite enclaves (*i.e.* the “microgranular mafic enclaves” described by Didier, 1973 and Didier *et al.*, 1982). Close to the strike-slip shear zone the Rouet-Plan de la Tour granite is deformed and exhibits a mylonitic texture developed under amphibolite facies conditions and progressively reworked under greenschist facies conditions (Onezime *et al.*, 1999; Corsini *et al.*, 2010). This granite could be a potential georesource for the pastes of Pendimoun, although the lack of cordierite, tonalite, microdiorite enclaves and amphibolite facies mylonites in the Pendimoun non-plastic particles allow to reject this hypothesis.

The Argentera-Mercantour massif (located 38km away from Pendimoun) outcrops at the French-Italian border and is part of the so-called “external crystalline massifs” of the Dauphinois-Helvetic zone of the Alps (Faure-Muret, 1955; Bogdanoff *et al.*, 1991; Von Raumer *et al.*, 2009). It is a stretched piece of the European Variscan orogenic crust extruded, and thus deformed, during Alpine continental collision (Bogdanoff *et al.*, 2000; Bigot-Cormier *et al.*, 2006; Lardeaux *et al.*, 2006; Schwartz *et al.*, 2007; Schreiber *et al.*, 2010). Initial detailed geological mapping (Faure-Muret, 1955; Malaroda *et al.*, 1970), followed by successive geological investigations, proposed to distinguish, in the Argentera-Mercantour massif, two main units (western and eastern unit) separated by a steeply dipping ductile shear zone, known as the “Ferriere–Mollières shear zone” or “Valletta shear zone” (Bogdanoff *et al.*, 1991; Musumeci and Colombo, 2002; Compagnoni *et al.*, 2010; Carosi *et al.*, 2016). High-resolution field mapping established firstly that abundant Variscan migmatitic paragneisses and orthogneisses outcrop in both western and eastern units (Gosso *et al.*, 2019), secondly that migmatites contain ante-migmatitic boudins of eclogites and high-pressure granulites (Latouche and Bogdanoff, 1987; Rubatto *et al.*, 2001 and 2010; Jouffray *et al.*, 2020) and thirdly that swarms of Permian-Triassic lamprophyres dykes intruded this Variscan basement (Filippi *et al.*, 2019 and 2020, fig. 5). Moreover, within the eastern unit, a late Variscan granitic pluton, called “Argentera Central Granite”, crosscut the pervasive migmatitic foliation (Boucarut, 1967; Ferrara and Malaroda, 1969; Corsini *et al.*, 2004). This pluton is composed of a two-micas granite, sometimes with leucogranitic facies in which muscovite is more abundant than biotite. It belongs to the very late Variscan Fe-rich granites well known in the “external crystalline massifs” of the Alps (Debon and Lemmet, 1999). This granite is intruded by abundant dykes of aplites, sometimes garnet-rich, or by dykes of lamprophyres and contains enclaves, centimetric to plurimetric in size, of sillimanite-bearing migmatites (fig. 6B). During Alpine orogeny, all these lithologies are metamorphosed under greenschist facies conditions (Corsini *et al.*, 2004; Lardeaux, 2014) while coeval alpine deformation is heterogeneously concentrated in mylonitic shear zones underlined by alternating small-sized recrystallised quartz (+/- albite) ribbons and muscovite+ chlorite rich layers (Corsini *et al.*, 2004; Sanchez *et al.*, 2010 and 2011).

In this tectonic system, the “Ferrière–Mollières shear zone” (*supra* fig. 5) is recognized as a transpressive shear zone active first in late Variscan times and later severely reworked during alpine collision (Musumeci and Colombo 2002; Corsini *et al.*, 2004; Sanchez *et al.*, 2011; Simonetti *et al.*, 2018). When intersected by this main regional-scale

shear zone, the “Argentera Central Granite”, its enclaves as well as the surrounding sillimanite-rich migmatites, are significantly deformed and exhibit a mylonitic texture developed under greenschist facies conditions (fig. 6A and C-D). Clearly the mylonitic “Argentera Central Granite”, with its associated dykes and surrounding migmatites, is a

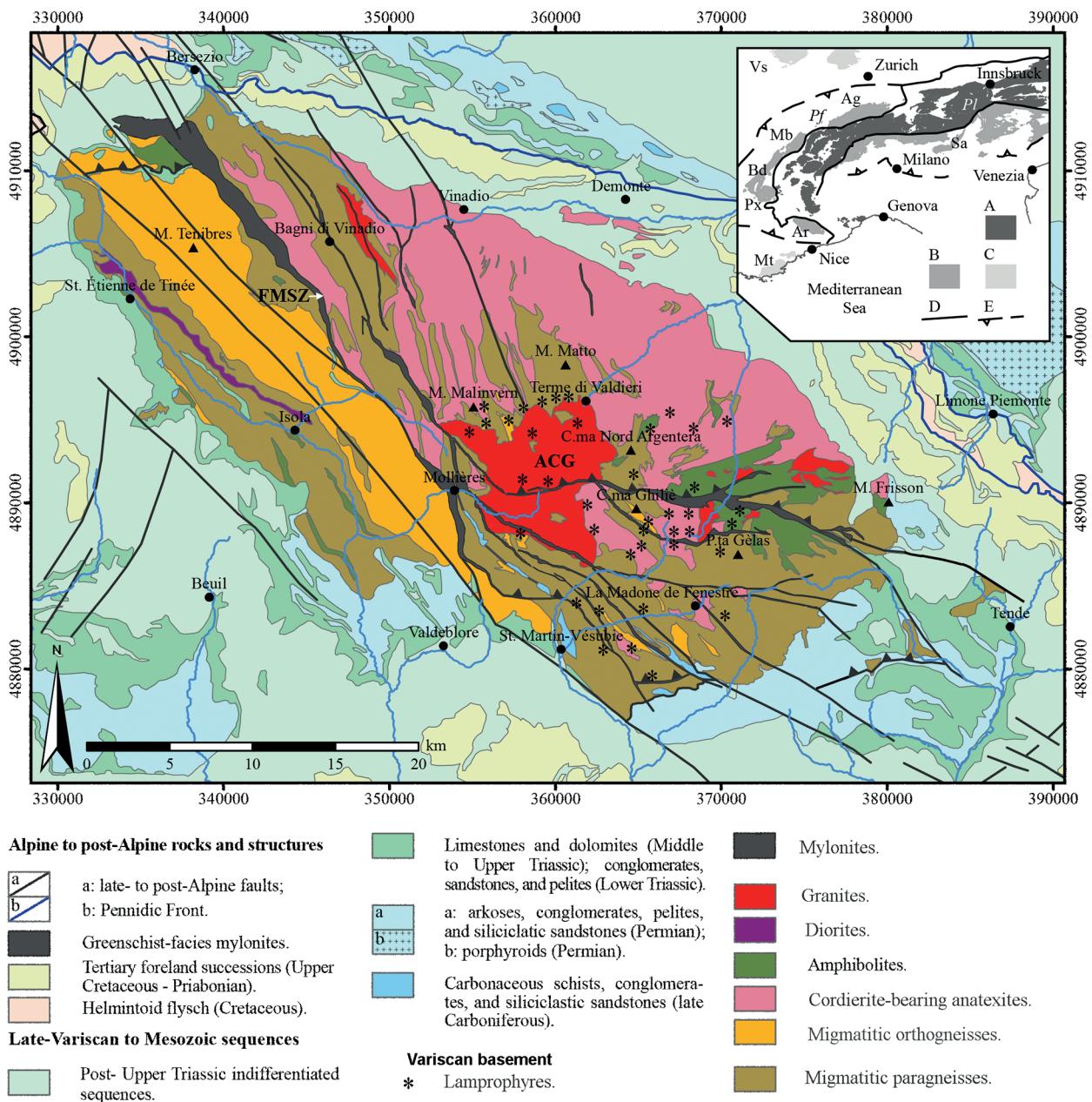


Fig. 5 – Geological map of the Argentera-Mercantour massif (modified after Goso *et al.*, 2019 and Filippi *et al.*, 2020). ACG : Argentera Central Granite ; FMSZ : Ferrière–Mollières Shear Zone. Note the heterogeneously distributed mylonitic shear zones. Box: Tectonic sketch of the Alps. A: Variscan basement rocks in the internal alpine zone. B: Variscan basement rocks of the external alpine zone (Ag: Aar-Gotthard Massifs, Ar: Argentera-Mercantour Massif, Bd: Belledonne Massif, Mb: Mont Blanc Massif, Px: Pelvoux Massif) and Southern Alps (Sa). C: Variscan basement rocks in Provence (Mt: Maures-Tanneron Massif). D: main tectonic structures delimiting the axial zone of the Alps (Pf: Penninic Front, Pl: Periadriatic Lineament). E: Alpine fronts.

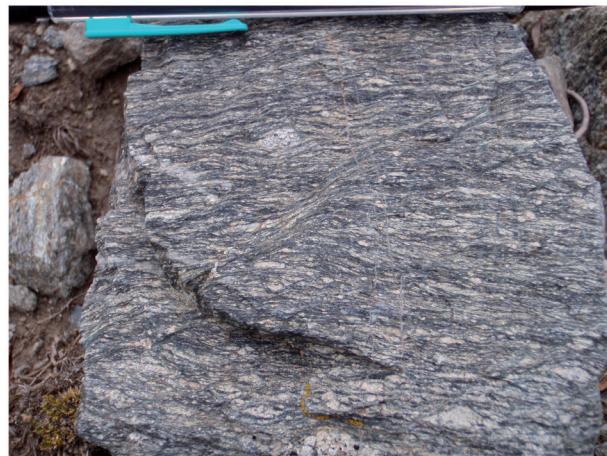
Fig. 5 – Carte géologique du massif de l'Argentera-Mercantour (modifiée d'après Goso *et al.*, 2019 et Filippi *et al.*, 2020). ACG : granite central de l'Argentera ; FMSZ : Shear Zone (zone mylonitique) de Ferrière–Mollières. Noter la distribution hétérogène des zones de cisaillement mylonitiques. Encadré : Schéma tectonique des Alpes. A : roches du socle varisque dans la zone interne des Alpes. B : roches du socle varisque dans la zone externe des Alpes (Ag : Aar-Gotthard, Ar : Argentera-Mercantour, Bd : Belledonne, Mb : Mont Blanc, Px : Pelvoux) et domaine Sud-Alpin (Sa). C : roches du socle varisque en Provence (Mt : Massif des Maures-Tanneron). D : principales structures tectoniques délimitant la zone axiale des Alpes (Pf : Front Pennique, Pl : Linéament Périadiquatique). E : fronts alpins.



A



B



C



D

Fig. 6 – Typical outcrops and mylonites in the Argentera-Mercantour Massif. A: mylonitic shear zone (in black) crosscutting the Argentera central granite (view from lac Nègre). B: intrusive contact of the Argentera central granite (light grey) within migmatites (dark brown) at the French-Italian border (Testa di Tablasse, view from Lago delle Portette); note the occurrence of numerous dark enclaves of migmatites in the granite. C: sample of a mylonitic granite (the dark colour results from abundant chlorite development in the fine-grained matrix); note highly deformed feldspars in the foliation plane. D: sub-vertical faults superposed on mylonitic zones promoting alteration of the Argentera-Mercantour formations.

Fig. 6 – Affleurements caractéristiques et mylonites du massif de l'Argentera-Mercantour. A : zone mylonitique (en noir) recoupant le granite central de l'Argentera (vue depuis le Lac Nègre). B : contact intrusif du granite central de l'Argentera (de couleur gris clair) et les migmatites (brun foncé) à la frontière franco-italienne (Testa di Tablasse, vue depuis le Lago delle Portette) ; noter les nombreuses enclaves sombres de migmatites dans le granite. C : échantillon de granite mylonitique (la couleur sombre est due à l'abondance de chlorite dans la matrice à grain très fin) ; noter la forte déformation des feldspaths dans le plan de foliation. D : fractures sub-verticales tardi-alpines superposées aux zones mylonitiques et favorisant l'altération des formations varisques du Massif de l'Argentera-Mercantour.

serious potential georesource for the Pendimoun ceramics as all the lithological/mineralogical components observed in the archaeological pastes are widespread in the geological target of interest.

If the production of the mixed pastes is also considered, it seems interesting to point out that the area within which the “Argentera Central Granite” and its surrounding migmatites are mylonised is located at less than 10km from the outcrops of the Mesozoic sedimentary cover rich in glauconite-bearing marls (*i.e.* early Cretaceous sediments, Faure-Muret *et al.*, 1956; Kerckhove 1969; Gosso *et al.*, 2019).

The Argentera-Mercantour massif, a potential source area: high-resolution geochemical analysis

In order to test the hypothesis of a potential source area located near the deformed edge of the “Argentera Central Granite” a comparative geochemical analysis of undeformed and unaltered granite samples, mylonitic granite samples, alteration deposits (alluvium and eluvium) from the investigated granite and potteries made from granitic pastes found at Pendimoun was carried out.

The geological samples were collected at the south-western margin of the “Argentera Central Granite”

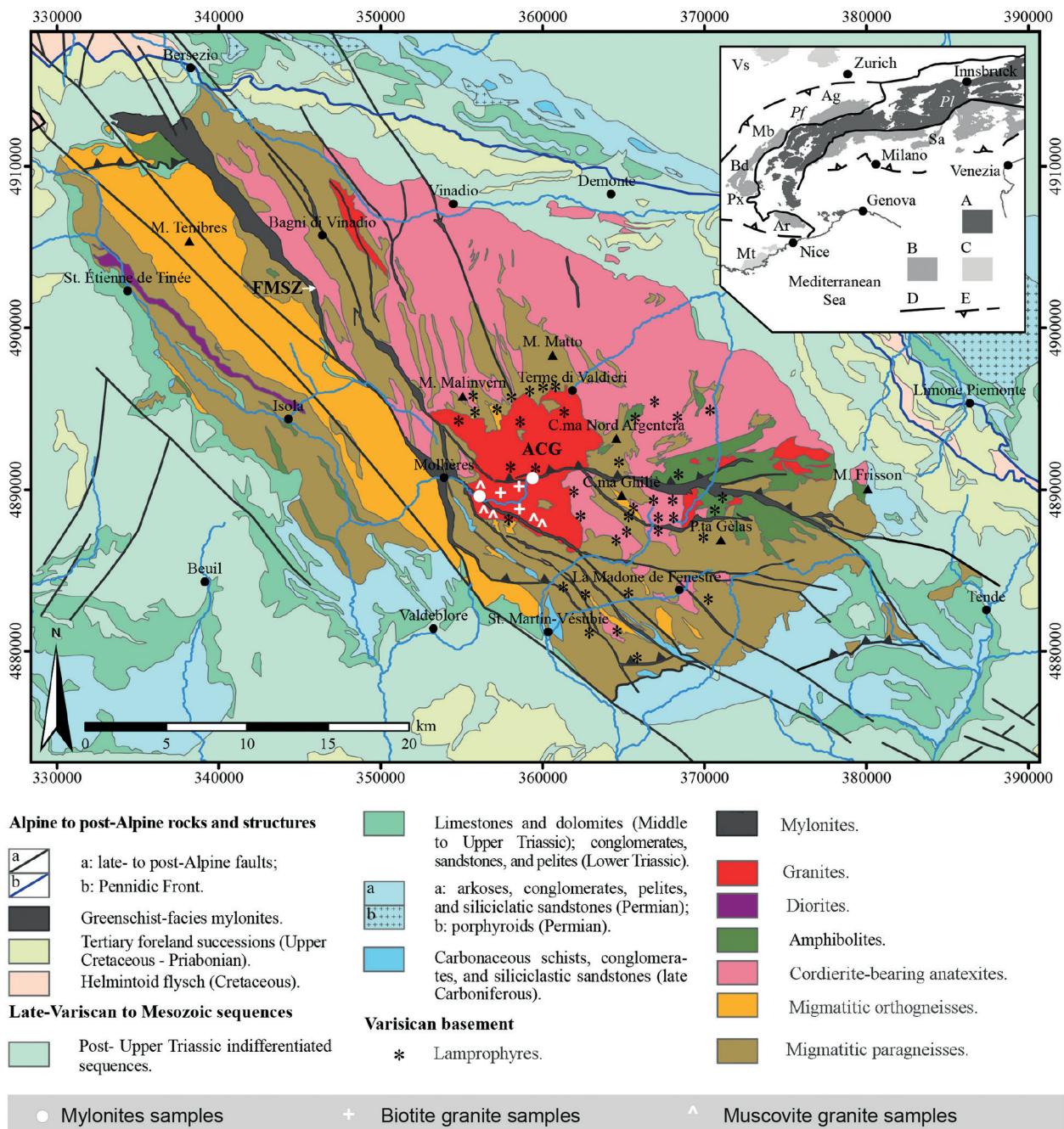


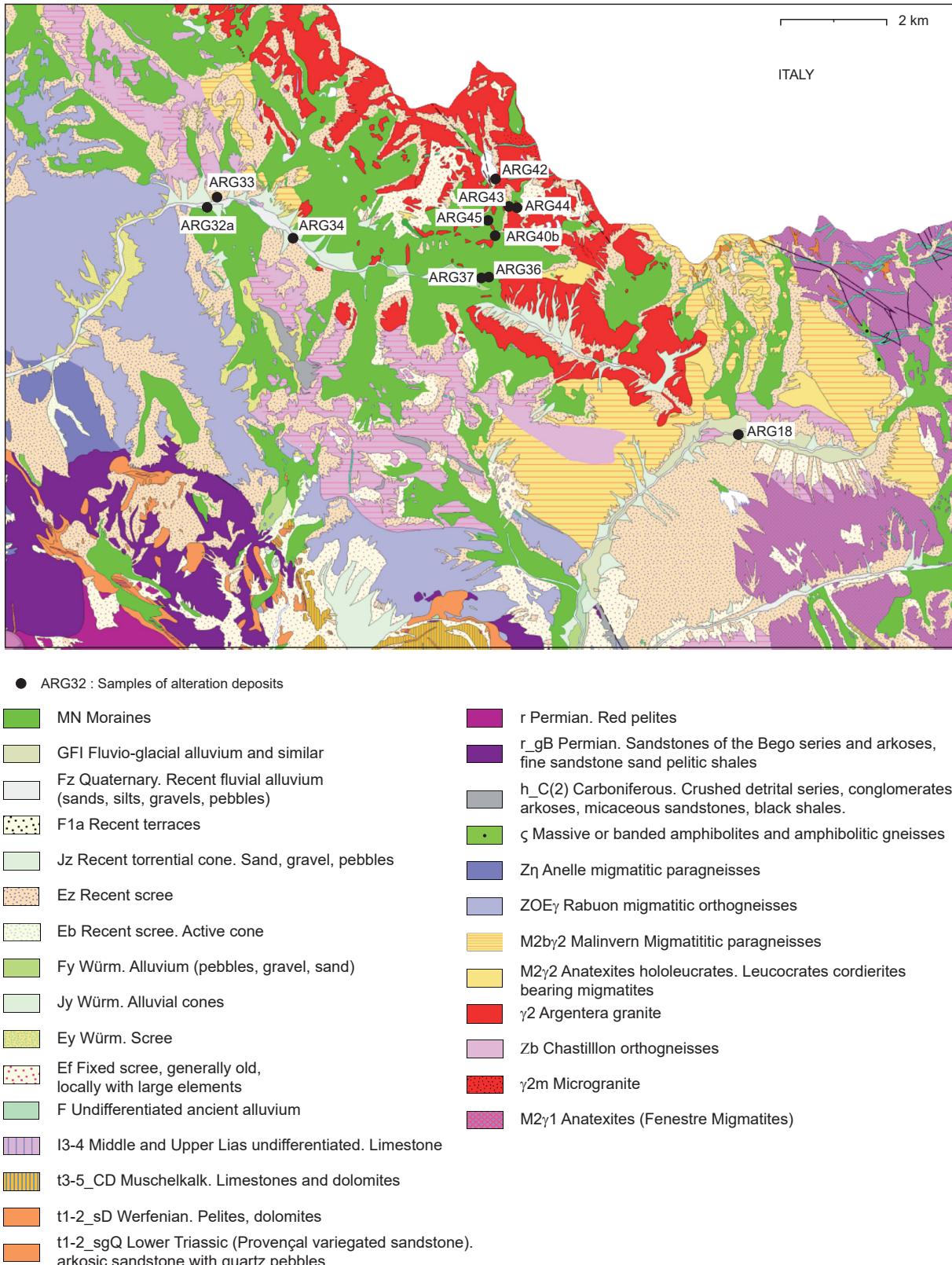
Fig. 7 – Location of the studied granitic and mylonitic rock samples from the Argentera central granite and used as references. Same map and legend as figure 5.

Fig. 7 – Localisation des échantillons de granite et de mylonite prélevés dans le granite central de l'Argentera et utilisés comme références. Carte et légende identiques à la figure 5.

between the Fremamorte crest and the Mollières valley where an efficient drainage network is well known (Ribolini and Spagnolo, 2008). Various petrographic facies of well-preserved granite, particularly two-micas leucogranite (with a very small amount of magmatic biotite) and two-micas granite (with 10 to 15% magmatic biotite in the mineral association) stemming from the Argentera-Mercantour were added in order to highlight the compositional variability of the underdeformed granitic protolith. Two additional samples of mylonitic granites were collected within the main regional scale

shear zone and a further eleven samples of alteration deposits downstream and upstream of the Salèse pass SD.1; fig. 7 and fig. 8).

Chemical analyses were performed using Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) and Inductively Coupled Mass Spectrometry (ICP-MS) for major and trace elements respectively at the Geochemical and Petrographical Research Center in Nancy (SARM laboratory, CNRS-CRPG; SD.2) following the procedure proposed by Carignan *et al.*, 2001).



From Geological map 1/50 000 vector harmonised (BRGM) Sheet N°1579 - Project : Alpes-Maritimes

Fig. 8 – Location of granitic and mylonitic alteration deposits samples (Tl) used as references in the high-resolution on the 1:50,000 vector harmonised geological map of the Argentera-Mercantour massif.

Fig. 8 – Localisation des échantillons de terres d'altération des roches granitiques et mylonitiques (Tl) utilisés comme références sur la carte géologique à haute résolution au 1/50 000 harmonisée du massif de l'Argentera-Mercantour.

Major element analyses

We first used the TAS diagram (*i.e.* Total Alkali vs. Silica), one of the most important diagnostic tools for the identification of magmatic rocks, based on bulk-rock chemical composition (Cox *et al.*, 1979; Le Maitre *et al.*, 2004).

This diagram (fig. 9) shows that the different facies of the unaltered “Argentera Central Granite” plot in the field of subalkaline granites, diagnostic for orogenic settings (*i.e.* continental collision granites, Barbarin, 1990). For comparison, the average chemical composition of this pluton (Debon and Lemmet, 1999) is also reported. The observed variation in chemical composition is related to changes in mineral composition (*i.e.* biotite abundance in particular) and thus reflects magmatic differentiation during granite genesis. With respect to these underformed and unaltered samples, the mylonitic

samples have depleted SiO₂ and to a lesser extent total alkali. This evolution is commonly described in granites mylonitised under greenschist facies conditions and reflects the destabilisation of K-feldspars, plagioclases and biotites, leading chlorite and white mica crystallisation, during deformation (Marquer, 1989; Streit and Cox, 1998; Rossi *et al.*, 2005). The alteration deposits display a similar chemical evolution trend, and, as regards two samples a stronger depletion of total alkali. The variability of their chemical compositions probably reflects different degrees of granite alteration. For comparison, the compositions of three samples of Pendimoun ceramics (mechanically cleaned) are also reported, even if this type of diagram is not really suited to pottery characterisation. Despite this difficulty, it can be noted that the compositions of archaeological ceramics are quite compatible with the compositional chemical trend shown by altered granite deposits.

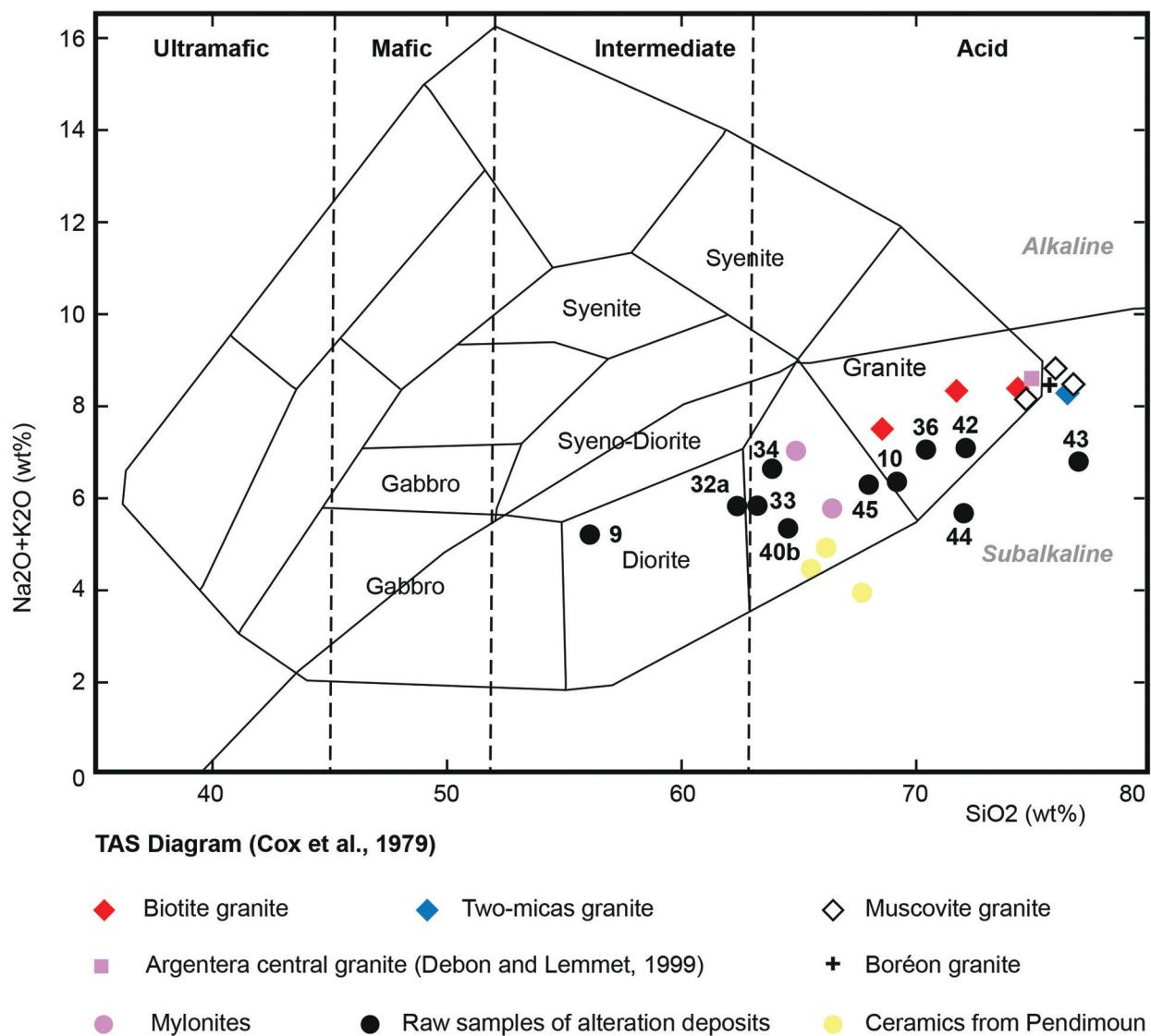


Fig. 9 – The geological samples from the Argentera-Mercantour and the archaeological samples from Pendimoun in the TAS (total alkali vs. silica) diagram.

Fig. 9 – Échantillons géologiques de l'Argentera-Mercantour et échantillons archéologiques de Pendimoun reportés dans le diagramme TAS (total alkalis vs. silice).

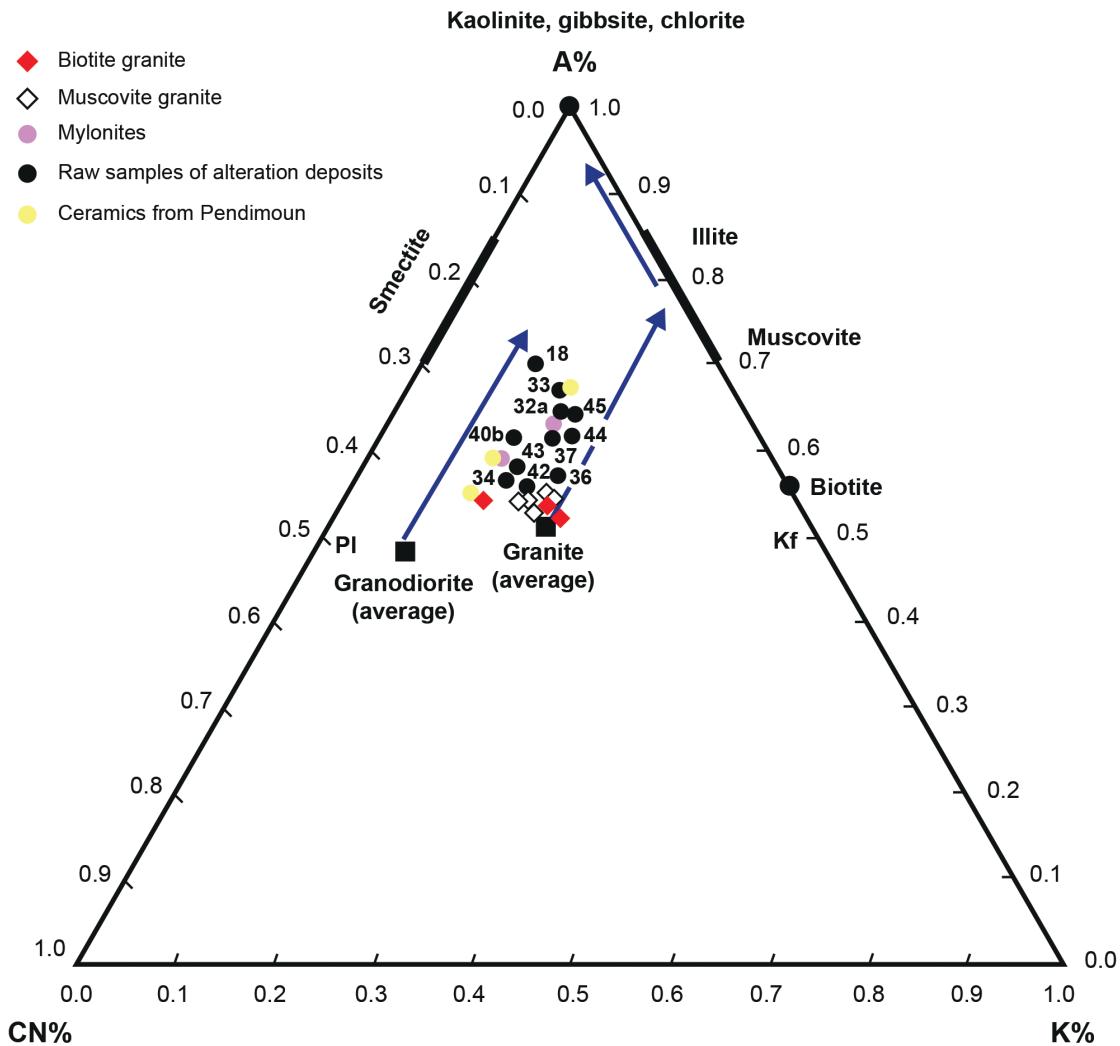


Fig. 10 – The geological samples from the Argentera-Mercantour and the archaeological samples from Pendimoun in the geochemical diagram of Nesbitt and Young (Nesbitt and Young, 1984).

Fig. 10 – Échantillons géologiques de l'Argentera-Mercantour et échantillons archéologiques de Pendimoun dans le diagramme géochimique de Nesbitt et Young (Nesbitt and Young, 1984).

Alteration or weathering of granitic rocks corresponds essentially to the hydrolysis of the major magmatic phases, following a commonly observed sequence including plagioclase, biotite, K-feldspar, muscovite and quartz from least to most stable phases (Goldich, 1938; Harris and Adams, 1966; Wilson, 1975; Nahon, 1991; Gerrard, 1994), although this expected susceptibility scale to weathering can be significantly altered by other factors such as climate and topography, or mineral microstructures (defects, dislocations, etc.) or naturally occurring soil organic acids and/or biological factors (see Wilson, 2004 for review with references therein). During progressive alteration major cations, particularly Na, Ca, K, Mg and Si, are released into solution during water/rock interactions while generally Al, Fe and Ti are less mobile and are therefore gradually concentrated in the weathering residues (Rice, 1973; Fritz and Ragland, 1980; Nesbitt *et al.*, 1980; Middelburg *et al.*, 1988). This general chemical mass balance testifies to the gradual formation of clay minerals by alteration of

feldspars into smectites and/or kaolinite ± gibbsite, of biotite into secondary chlorite, smectites and/or kaolinite or gibbsite and muscovite into illite, smectites and/or kaolinite ± gibbsite (Millot, 1964; Velde, 1984; Nahon, 1991; Gerrard, 1994; Sequeira Braga *et al.*, 2002). Thus, an efficient tool to evidence and quantify the chemical alteration trends of granitic rocks is the diagram established by Nesbitt and Young, 1984 based on experimentally determined alteration rate constants of minerals from plutonic rocks.

In this diagram (fig. 10), the commonly observed alteration trends for granitic and granodioritic rocks are well evidenced (blue arrows). With respect to undeformed and unaltered samples, the mylonitic samples and the alteration deposits follow a similar chemical trend, which is typically described during progressive alteration of granitoïds. Nevertheless, the most remarkable observation is the compatibility of the analyses obtained on the studied archaeological ceramics with those of the deformed and altered “Argentera Central Granite” samples.

Trace element analyses

We also carried out trace element analysis on the bulk ceramic samples and granite-derived alteration deposits. In a continental crust normalised trace-element spider

diagram (normalisation values from McDonough and Sun, 1995) the Pendimoun ceramics and the altered granite deposits display compatible, quite similar for some natural samples, spectra with a notable negative Eu anomaly and similar slight Sm and Yb anomalies (fig. 11 and fig. 12).

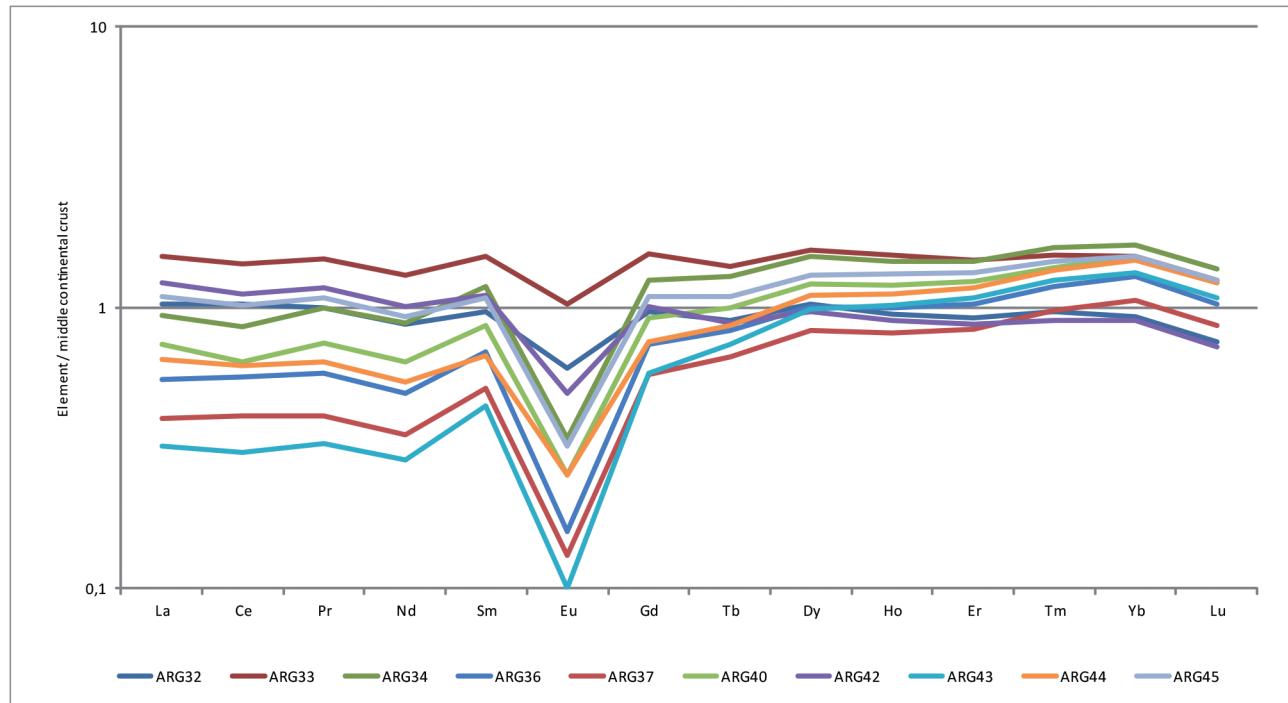


Fig. 11 – Trace-element spider diagram, normalized to continental crust values (after McDonough and Sun, 1995), of reference samples from geological contexts in the Argentea-Mercantour massif.

Fig. 11 – Diagramme des éléments-traces, normalisé aux valeurs de la croûte continentale (d'après McDonough and Sun, 1995), des échantillons géologiques de référence de l'Argentera-Mercantour.

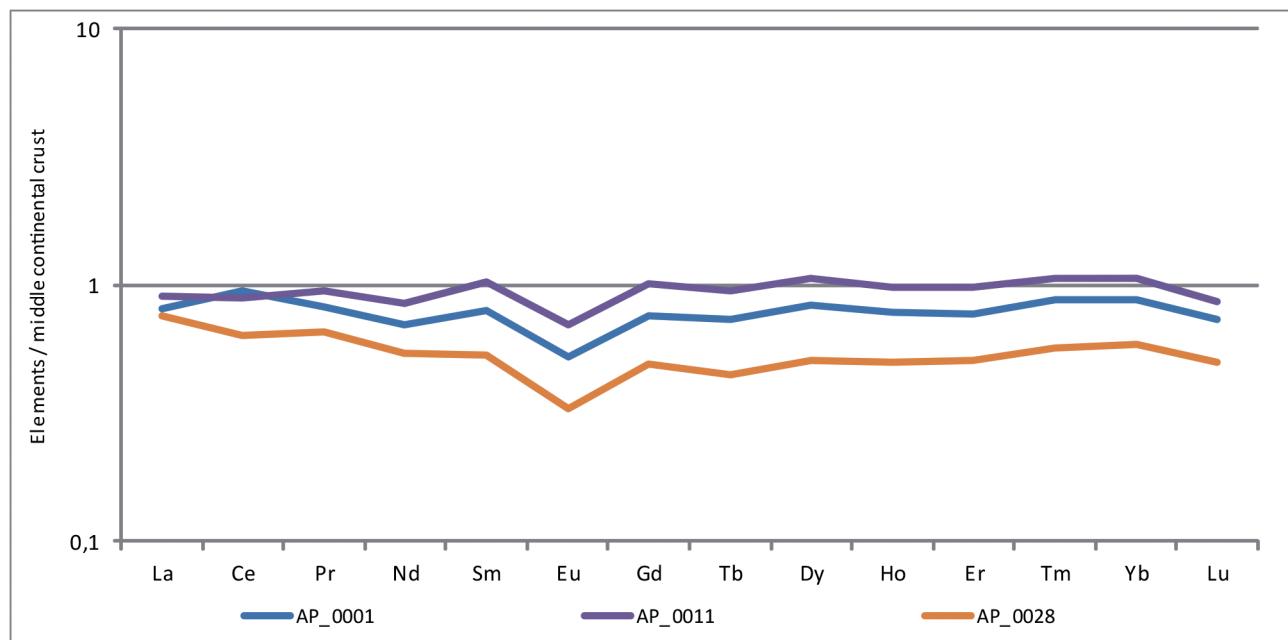


Fig. 12 – Trace-element spider diagram, normalized to continental crust values (after McDonough and Sun, 1995), of the archaeological samples from Pendimoun.

Fig. 12 – Diagramme des éléments-traces, normalisé aux valeurs de la croûte continentale (d'après McDonough and Sun, 1995), des échantillons archéologiques de Pendimoun.

Trace elements spectra from ceramic samples match altered “Argentera Central Granite” samples, supporting the latter as potential source for the pottery of Pendimoun (fig. 12).

Remaining issues

Detailed comparative and multi-analytical investigations (*i.e.* mineralogical, petrographic, microstructural and geochemical analyses) indicate that the most likely source of raw materials for the studied ceramics are the mylonitised and altered zones of the “Argentera Central Granite” and its related rocks.

Although this working hypothesis is particularly robust, it nevertheless raises a significant number of questions:

- In a high-altitude topographical context, with climatic conditions that are not favourable to the action of biological factors, mechanical erosion is efficient but chemical weathering of granitic rocks is limited. In such a context, how can the significant production of clay-rich alteration deposits be explained?
- The “Argentera Central Granite” mylonites are particularly rich in chlorite resulting from the destabilisation of biotites. In this case, why is chlorite so scarce as regards the pottery sample?
- The “Argentera Central Granite” is closely associated with sillimanite-bearing migmatitic paragneisses and garnet-rich aplites. Why were only extremely rare sillimanite or garnet grains observed in potteries recovered from Pendimoun?

ALPINE DEFORMATION AND NATURAL ALTERATION CYCLE OF ROCKS FROM THE ARGENTERA-MERCANTOUR MASSIF: CONSEQUENCES FOR RAW MATERIALS PRODUCTION

The main imprint of Alpine continental collision in the Argentera-Mercantour Massif is the development of a regional-scale network of greenschist facies ductile shear zones resulting in anastomosed mylonitic corridors (*supra* fig. 5 and fig. 6). These mylonites, identified as early as the pioneering work carried out by Faure-Muret (Faure-Muret, 1955), have undergone numerous detailed mineralogical and structural studies (Malaroda *et al.*, 1970; Musumeci and Colombo 2002; Corsini *et al.*, 2004; Sanchez, 2010; Sanchez *et al.*, 2010 and 2011; Simonetti *et al.*, 2018). From a mineralogical point of view, biotites and feldspars are the heaviest transformed minerals during syn-metamorphic deformation in mylonitic granites. Primary magmatic phases are replaced by white micas (muscovite and/or sericite) and chlorite (see Sanchez *et al.*, 2011 for review). Under surface conditions and in the presence of water, chlorite neo-formation at the expense of primary biotites facilitates the formation of clay minerals such as interstratified chlorite-smectite and

pure smectites, whereas white micas are easily altered into interstratified mica-illite and illite/smectite species (Velde, 1984). All these initial neo-formed clay phases can subsequently evolve into kaolinite (Millot, 1964; Velde, 1984; Nahon, 1991). Moreover, in mylonites the very small size of the recrystallised grains (fig. 6B) increases the water/grain contact surfaces and increases the kinetics of the hydrolysis reactions. In addition, the presence of planar discontinuity (schistosity planes, shear bands, micro-cracks) facilitates the circulation and trapping of water and thus hydrolysis reactions.

Taking these data into account, the mineralogical composition of the eleven samples of alteration deposits was analysed (*supra* fig. 8 and fig. 13). These samples all contained fragments of mylonitic rocks and numerous plastically deformed quartz grains (fig. 14AD). The systematic presence of chlorite, a great amount of muscovite, and the presence of clay minerals could be stated. Some samples contain also relics of magmatic K-feldspars. These were analysed and dated by the ^{40}Ar - ^{39}Ar method at the Géoazur laboratory (Sophia-Antipolis). Ages were calculated using the ArArCALC-software (Koppers, 2002). Analytical procedures and raw data can be downloaded from supplementary materials (SD.3). $^{40}\text{Ar}/^{39}\text{Ar}$ age spectra are presented in figure 15. K-feldspar grains yield an age of reopening of the isotopic system at 19+/-2 Ma, *i.e.*, the age of alpine deformation, thus confirming the formation of these clay soils at the expense of the mylonites.

All in all, these mineralogical and microstructural features indicate that mylonites may be considered as being precursors to chemical alteration or weathering and explain, even in the absence of abundant vegetal cover, the production of clay-rich alteration deposits, and thus raw materials for granitic pastes, at high altitudes. In other words, mylonite zones are “accelerators” of the formation of clay-rich alteration soils.

Furthermore, when migmatitic paragneisses or garnet-bearing aplites are deformed within greenschist facies shear zones, sillimanites are severely transformed into white micas and garnets into chlorites. Because the raw materials used for the pottery of Pendimoun come from these deformation zones, sillimanite or garnet grains are rarely observed in these ceramics.

THE ROLE OF THE FIRING CYCLE ON THE EVOLUTION OF RAW MATERIALS: CONSEQUENCES FOR THE CHARACTERISATION OF NEOLITHIC POTTERY

Mineralogy of experimentally fired clay-rich alterites from the Argentera-Mercantour massif

The sampled Argentera-Mercantour clay-rich alteration soils were used to produce experimental ceramics in the form of tablets of standard dimensions (c. 5x5

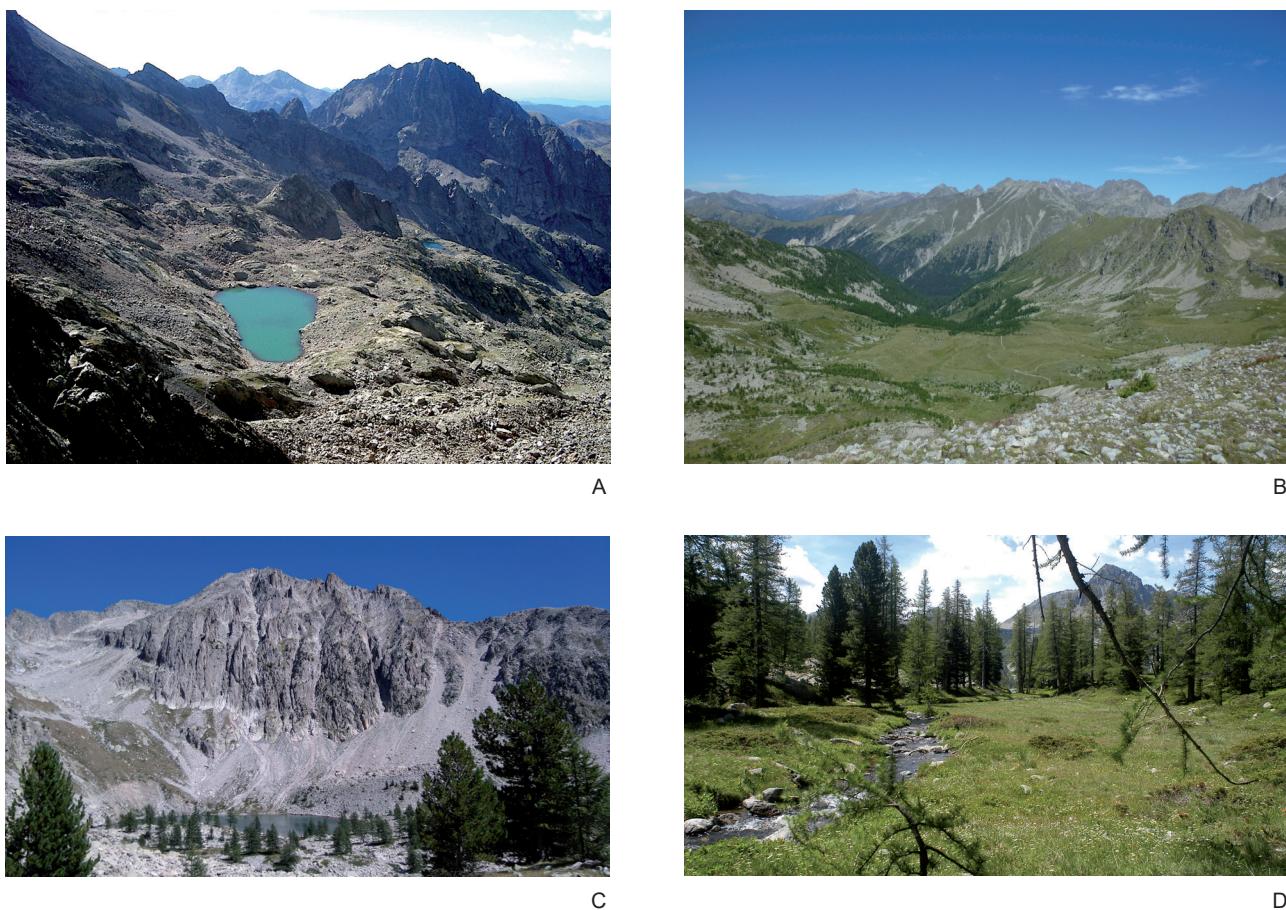


Fig. 13 – Present-day areas of granitic / mylonitic alteration deposits in the Argentera-Mercantour massif. A: Riofreddo lakes (Italy); B: Prals valley (France); C: Tavels lake (France); D: Mollières valley (France).

Fig. 13 – Zones actuelles de dépôt des terres d'altération granitiques / mylonitiques dans le massif de l'Argentera-Mercantour. A: lacs de Riofreddo (Italie) ; B : vallon de Prals (France) ; C : Lac de Tavels (France) ; D : vallée de Mollières.

cm) fired at different temperatures (300°C, 500°C and 700°C) within a controlled and specific environment (see experimental procedures in SD.4 to SD.9). A series of thin sections were made from these experimental potteries in order to determine their specific mineralogy (*supra* fig. 14EF and fig. 16).

The observations of the thin sections carried out under a polarised microscope revealed the following features that characterise the mineralogical change dependent on the temperature measured in the kiln:

- At 300°C no significant change in the mineral composition of the alteration deposits was observed. Clay minerals are stable, chlorite and muscovite are abundant, quartz grains are in textural equilibrium with the matrix and relicts of primary magmatic phases such as large-sized biotites and feldspars are observable and retain their optical characters and their initial morphology.
- From 500°C, most of the clay minerals have disappeared whereas chlorites are heterogeneously preserved. Indeed, some chlorites, only partly transformed into biotite, were still observed. These incomplete mineral transformations attest for

disequilibrium conditions controlled by kinetic factors. The matrix contains neo-formed and small-sized muscovite, biotite, quartz, and ilmenite which demonstrate the growth of new mineral phases and thus the setting up of modal (*i.e.* discontinuous) reactions. These new grains of biotite show a texture and sizes very different from those of conserved magmatic biotites, which preserve their original birefringence and pleochroism. Primary muscovite, quartz, and feldspars are observed, with their optical characteristics still well preserved.

- At 700°C chlorites have completely disappeared and the experimental potteries were characterised by a fully recrystallised, brownish-reddish, biotite-rich matrix. Reaction rims, of variable sizes, occur at boundaries between primary quartz and/or feldspar grains and matrix as well as within internal mineral micro-fractures (fig. 16). They consist mainly of new grains of biotite, quartz, ilmenite, rare white micas and their heterogeneous development is, here too, indicative of kinetically controlled discontinuous mineral reactions. At that temperature, primary magmatic mica grains retain their original optical characters.

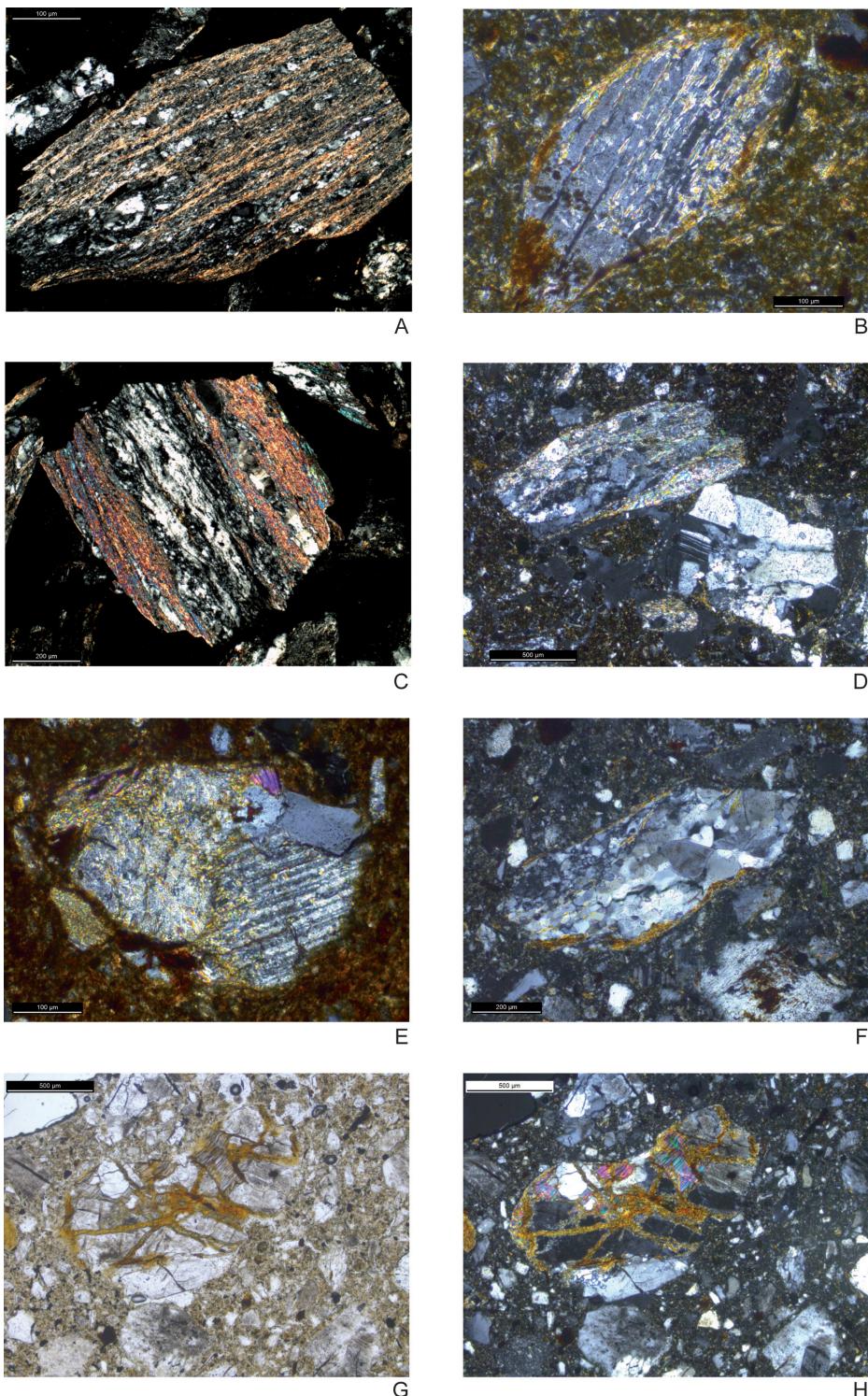


Fig. 14 – Mineral assemblages and microstructures in Argentera-Mercantour's granitic and mylonitic alteration soils and in experimental pastes. A: fragment of mylonite in alteration soil (crossed polars); B: plagioclase, partly replaced by micas, in alteration soil (crossed polars); C: fragment of mylonite, with ribbon of fully recrystallized quartz, in alteration soil (crossed polars); D: lithoclasts of deformed granitic rocks in alteration soil (crossed polars); E: experimental ceramic showing partial replacement of feldspar by micas within a biotite rich matrix (crossed polars); F: experimental ceramic for observing a corona of secondary biotite around a recrystallized quartz-bearing aggregate (crossed polars).

Fig. 14 – Assemblages minéralogiques et microstructures dans les terres d'altération granitiques et mylonitiques de l'Argentera-Mercantour et dans les pâtes expérimentales. A : fragment de mylonite une terre d'altération (lumière polarisée analysée) ; B : plagioclase partiellement altéré en micas dans une terre d'altération (lumière polarisée analysée) ; C : fragment de mylonite avec ruban de quartz recristallisé dans une terre d'altération (lumière polarisée analysée) ; D : lithoclastes granitiques déformés avec grains de quartz recristallisés dans une terre d'altération (lumière polarisée analysée) ; E : céramique expérimentale montrant la transformation partielle des feldspaths en micas au sein d'une matrice riche en biotite (lumière polarisée analysée) ; F : céramique expérimentale montrant une auréole de réaction à biotite autour d'un agrégat de quartz recristallisé (lumière polarisée analysée).

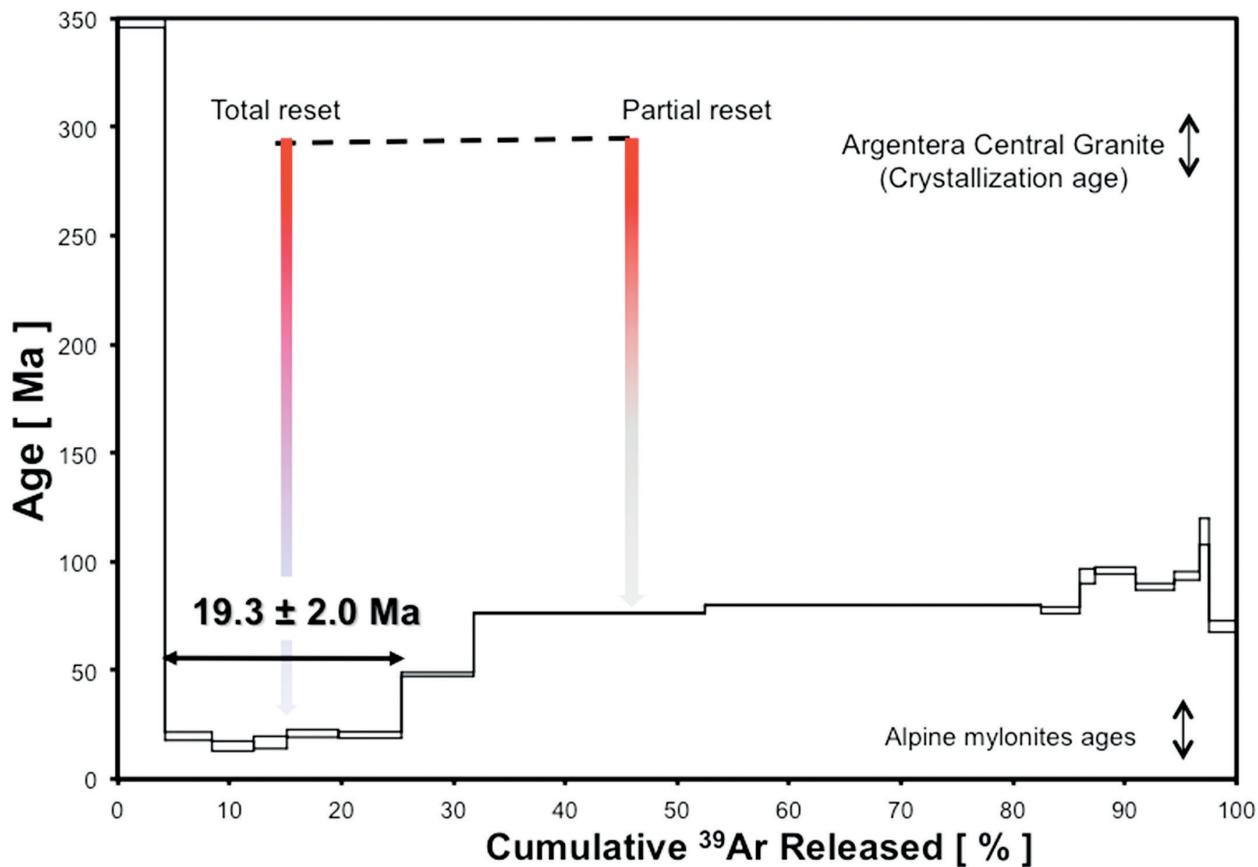


Fig. 15 – $^{40}\text{Ar}/^{39}\text{Ar}$ age spectra of a sample (ARG 45, fig. 8) from alteration deposits in the Argentera-Mercantour Massif. The age of the Argentera central granite is indicated showing the chemical re-opening of the analysed magmatic K-feldspar. The total reset (10% of ^{39}Ar released) yields the age of end of diffusion in the crystal, which corresponds to the age of alpine mylonitic zones development.

Fig. 15 – Spectres des âges $^{40}\text{Ar}/^{39}\text{Ar}$ pour un échantillon (ARG 45, fig. 8) des terres d’altération du massif de l’Argentera-Mercantour.
L’âge du granite central de l’Argentera est indiqué démontrant la réouverture chimique du feldspath potassique magmatique analysé.
La remise à zéro totale (10 % de ^{39}Ar relâché) marque l’arrêt de la dernière diffusion dans le cristal et correspond à l’âge du développement des zones mylonitiques alpines.

Comparison with available petrologic and thermodynamic databases

The analogies established between reaction mechanisms acting during industrial pottery production by firing of clay materials and during metamorphism of natural rocks over geological times have been emphasised for several decades (Peters and Iberg, 1978; Maggetti, 1982; Riccardi *et al.*, 1999). Therefore, the constraints brought by phase equilibrium diagrams from both “industrial and natural metamorphism” are often used to better understand the processes of ancient pottery production (Maggetti and Heumann, 1979; Maggetti, 1981; Heumann, 1989; Duminico *et al.*, 1995; Binder *et al.*, 2018). More particularly, the quantitative results obtained for the temperatures suffered during low-pressure metamorphism (*i.e.* contact metamorphism) by clay-rich sedimentary rocks can be used to test the robustness of our experimental results and to better interpret the pottery production discovered at Pendimoun (see Spear, 1993 and Bucher and Frey, 2002, with references therein for a general review of the available petrologic datasets).

In meta-sediments, under low-pressure conditions, the upper stability limit of clay minerals is around 250–280°C. Depending on its chemical composition, chlorite is stable under a large temperature range between 250 and 500°C. The thermal stability of micas depends on the amount of free water present in the chemical system under consideration. However, for muscovite the uppermost stability condition never exceeds 650°C for pressure lower than 1kbar, while for biotite the maximum stability conditions can reach 800°C. Moreover, the evolution of low-temperature metamorphic rocks (*i.e.* T < 600°C) is governed by disequilibrium conditions characterised by incomplete reactions and different sub-systems controlled by the kinetics of reactions.

However, in metamorphosed meta-sediments the P-T stability fields of the various phases are also dependent on the natural chemical system (*i.e.* the specific bulk-rock chemistry). Consequently, in order to highlight the importance of the chemical composition of the studied alterites on mineral stability conditions thermodynamic modelling was performed on the basis of two specific samples (ARG34 and ARG32) using the THERIAK-DOMINO

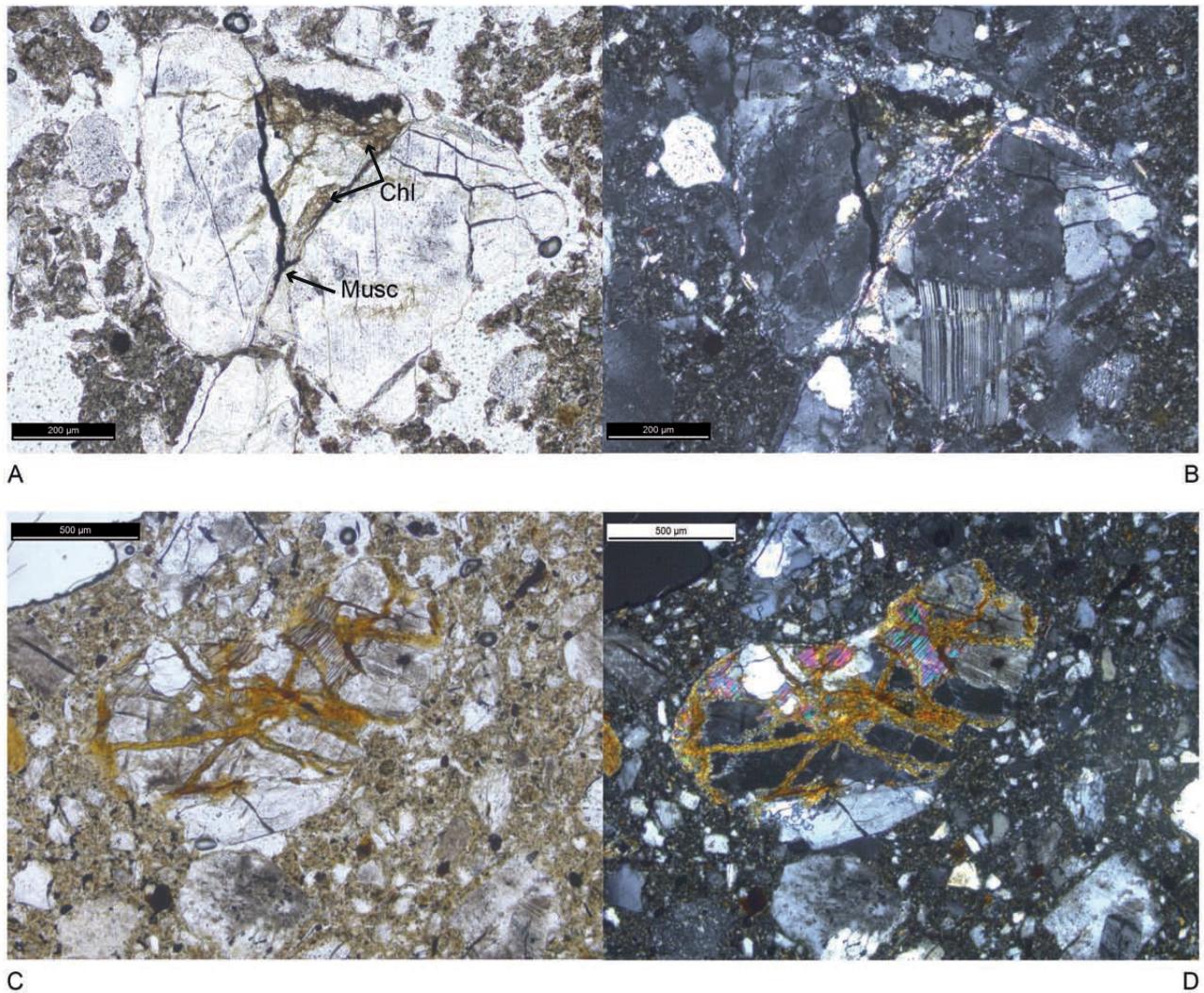


Fig. 16 – Comparison of mineral assemblage in the same raw and fired (experimental ceramic) alteration earth highlighting the transformation of feldspar into biotite and muscovite. A: note the crystallization of new phases (micas) at grain rims and within micro-cracks of feldspar (parallel polars); B: same as A with crossed polars.

Fig. 16 – Comparaison des assemblages minéralogiques dans le même échantillon de terre d'altération crue et cuite (céramique expérimentale) montrant la transformation des feldspaths en biotite et muscovite. A: on remarquera la cristallisation des phases néoformées (micas) aux joints de grains et dans les micro-fractures des feldspaths (lumière polarisée non analysée) ; B : idem, C en lumière polarisée analysée.

method (De Capitani and Petrakakis, 2010) and the Holland and Powell database (Holland and Powell, 2004). This method, similar to other mineral equilibrium modelling methods (such as THERMOCALC, Powell *et al.*, 1998), allows us to decipher the effects of all major chemical elements from a given “real bulk-rock composition” on the stability of the observed minerals. THERIAK-DOMINO is a free energy minimisation programme, taking into account mixing models for mineral solid solutions and enabling calculation of isochemical phase diagrams. This technique explores not only the P-T stability fields of mineral associations (including the relative abundances of the different phases in a given mineral assemblage) but also the effects of the Fe-oxidation state or the influence of the amount and nature of fluids (H_2O , CO_2) on the stability field of a given mineral association. In this study, the evolution of the two samples

was modelled in the SiO_2 - Al_2O_3 - TiO_2 - FeO - MgO - MnO - CaO - Na_2O - K_2O - P_2O_5 - H_2O system. H_2O in excess was considered to be consistent with the generally accepted pottery production processes. Considering archaeological samples, the iron oxidation state at the time of ceramics production is difficult to estimate. Therefore, we produced a model with total iron considered FeO as a first, simple, approximation. Because in this study, the temperature is the only variable of interest, only the THERIAK programme was used in order to depict the mineralogical evolution through a progressive temperature increase from 200°C up to 700°C.

In the ARG34 sample, the following most thermodynamically stable mineral associations were obtained:

- At 200°C: Chlorite-Illite-Quartz-Zeolites-Titanite (<1%). Hydrated minerals include 34.1% chlorite, 36.7% illite and 29.2% zeolites.

- At 250°C: Chlorite-Muscovite-Quartz-Feldspar-Zeolites-Titanite (<1%). Hydrated phases include 31.8% chlorite, 40.9% muscovite and 27.3% zeolites.
- At 300°C: Chlorite-Muscovite-Quartz-Feldspar-Zeolites-Ilmenite (<1%). Hydrated minerals include 36.3% chlorite, 40.9% muscovite and 22.8% zeolites.
- At 350°C: Chlorite-Muscovite-Quartz-Feldspar-Biotite-Ilmenite (<1%). Hydrated phases include 10% chlorite, 55% muscovite and 35% biotite.
- At 400°C: Muscovite-Quartz-Feldspar-Biotite-Ilmenite (<1%). Hydrated minerals include 58.7% muscovite and 41.3% biotite.
- At 450°C: Muscovite-Quartz-Feldspar-Biotite-Ilmenite (<1%). Hydrous phases include 52.9% muscovite and 47.1% biotite.
- At 500°C: Muscovite-Quartz-Feldspar-Biotite-Ilmenite (<1%). Hydrous minerals include 39.7% muscovite and 60.3% biotite.
- At 550°C: Muscovite-Quartz-Feldspar-Biotite-Ilmenite (<1%). Hydrous phases include 10.3% muscovite and 89.7% biotite.
- In the range 560-600°C: Biotite-Quartz-Feldspar-Cordierite-Ilmenite (<1%).
- At 650°C: Quartz-Cordierite -Feldspar-Ilmenite (<1%).
- At 700°C: Quartz-Feldspar-Cordierite-Sanidine-Ilmenite (<1%).

In the ARG32 sample, the following most thermodynamically stable mineral associations were obtained:

- At 200°C: Chlorite-Illite-Quartz-Feldspar-Zeolite. Hydrated minerals include 44.6% of chlorite, 49.5% illite and 5.9% zeolite.
- At 250°C: Chlorite-Muscovite-Quartz-Feldspar-Zeolite-Titanite (1%). Hydrous phases include 41.7% chlorite, 54.5% muscovite and 3.8% zeolite.
- At 300°C: Chlorite-Muscovite-Quartz-Feldspar-Zeolite-Titanite (0.5%)-Ilmenite (0.4%). Hydrous minerals include 41.9% chlorite, 55.1% muscovite and 3% zeolite.
- At 350°C: Muscovite-Biotite-Quartz-Feldspar-Ilmenite (<1%). Hydrous phases include 62.2% muscovite and 37.8% biotite.
- At 400°C: Muscovite-Biotite-Quartz-Feldspar-Ilmenite (<1%). Hydrous phases include 58% muscovite and 42% biotite.
- At 450°C: Muscovite-Biotite-Quartz-Feldspar-Ilmenite (<1%). Hydrous phases include 53.1% muscovite and 46.9% biotite.
- At 500°C: Muscovite-Biotite-Quartz-Feldspar-Ilmenite (<1%). Hydrous phases include 93.9% biotite and 6.1% muscovite.
- At 530°C: Muscovite-Biotite-Quartz-Feldspar-Ilmenite (<1%). Hydrous phases include 97.8% biotite and 2.2% muscovite.
- In the range 550-650°C: Biotite-Quartz-Feldspar-Cordierite-Ilmenite (<1%).
- At 700°C: Quartz-Feldspar-Cordierite-Ilmenite (<1%).

With respect to the general constraints extracted from metamorphosed meta-sediments, it must be stressed that chlorite is stable under a more restricted temperature range of 250-350°C in the ARG34 sample and of 200-300°C in the ARG32 sample. The association of muscovite and biotite is only stable between 400 and 550°C in the ARG34 sample and between 350-530°C in the ARG32 sample. Muscovite stability never exceeds 550°C, while ilmenite is an accessory phase under a large temperature range between 300 and 700°C. These results are clearly consistent, at the first order, with the mineralogical evolution deduced from our experiments. Indeed, at 300°C chlorite and muscovite are both stable, while at 500°C the diagnostic association is muscovite + biotite + quartz + ilmenite. The occurrence of some clay minerals at 300°C or of relicts of chlorites at 500°C are controlled by kinetic factors (*i.e.* short duration of experiments) allowing some reactants to be preserved (incomplete reactions).

Implications for the Pendimoun pottery

Under the polarised microscope, the Pendimoun ceramics display reddish to light-brown matrix composed of small-sized neo-formed biotites, muscovites and minor quartz and ilmenite (*supra* fig. 3) Thin reaction coronas are commonly observed at grain boundaries of primary magmatic quartz and feldspars (*supra* fig. 3A). In these reaction sites quartz, muscovite and biotite are typically the neo-formed phases. First, these data demonstrate that maximal temperatures of 550°C were reached during firing. Second, the potteries from Pendimoun were potentially produced within the temperature range of between 350 and 550°C. This explains why – even if the original raw materials were rich in chlorite – the latter is absent from the pottery sample as a result of firing conditions. Moreover, because in the various reaction sites (new grains in the matrix, coronas around primary magmatic grains) the proportions of biotites are always significantly higher than those of muscovites (*supra* fig. 3B and D), the most probable firing temperatures were probably close to 500-550°C. Nevertheless, these firing conditions also explain the excellent preservation of the primary magmatic phases in the ceramics, these minerals being thermodynamically metastable under these temperatures. Furthermore, a temperatures range of about 200°C accounts for the existence of the very likely thermal gradients that would exist in the types of furnaces used for pot production during Neolithic times. Even with such a thermal gradient, the mineralogy of the ceramics remained qualitatively constant.

Finally, it should be noted that the proposed temperature range is compatible with the preservation, in archaeological ceramics, of optical characteristics (birefringence and pleochroism) on both biotite and muscovite that tend to disappear for temperatures above 800°C (Riccardi *et al.*, 1999).

DISCUSSION AND PROSPECTS

In this study Neolithic “granitic” pottery pastes (17 samples) were compared to five possible geological sources. Geochemical, mineralogical, petrographic and textural investigations enable us to propose that mylonitic and altered meta-granites from the Argentera-Mercantour Massif are the most likely source of raw materials for the studied ceramics. Eleven alteration deposits from this area were compared with the pottery samples recovered from the archaeological site and subjected to firing experiments and thermodynamic modelling.

The main geological sources for raw materials used to produce “granitic pastes” are quartzo-feldspathic aluminium-rich rocks, thus granites and orthogneisses or paragneisses (*i.e.* dominant lithology in the continental crust). In the gneisses, high-temperature minerals are frequent, particularly aluminium silicates (andalusite/sillimanite/kyanite) and garnets. However, during exhumation and cooling of the rocks and later alteration (*i.e.* “natural cycle”) these minerals are easily replaced by chlorite, muscovite and illite particularly in severely deformed zones (mylonites). Consequently, even when high-temperature metamorphic phases are not observed in micaceous, quartzo-feldspathic, pots, a gneissic source for the considered raw materials cannot be definitely ruled out. Moreover, during the cooling and alteration of granites biotite is easily replaced by chlorite, while muscovite remains much more stable. Thus, even when muscovite is largely the dominant mica in a given ceramic, a biotite-rich granite is still a possible geological source. When it comes to “granitic pastes”, detailed mineralogical and textural investigations must be combined with high-resolution geochemical data, including trace elements spectra, to conduct robust sourcing.

During the firing of raw materials, clay minerals and chlorite are replaced by muscovite and biotite for temperatures above 350–400°C. At around 500°C, abundant new grains of biotite, associated with minor amounts of muscovite, quartz and Fe-oxydes, crystallise leading the formation of the so-called light-brown and biotite rich matrix. Contrary to previous interpretations (Échallier in Binder, 1991), this type of matrix is not composed by a felting of primary biotites, but by the crystallisation of fine-sized biotite new grains during firing. Furthermore, during the “industrial cycle” clay minerals as well as chlorites react and are replaced by new grains of micas, quartz and feldspars. This gradual disappearance of the clay minerals explains the very low level of small pores (*i.e.* <0.1 µm) observed thanks to Hg intrusion porosimetry at a low level which is itself at the origin of a poor preservation of lipids within this type of pottery fabric (Drieu *et al.*, 2019). Consequently, in archaeological ceramics produced by the firing of “granitic pastes” the amount of clayey minerals initially present in the raw materials should be dramatically under-estimated. Thus, the question about the workability of such kind of pastes (Aissaoui, 2018) needs to be largely reconsidered.

As previously suggested (Gabriele, 2014), the hypothesis of the collection of soils, used for the pottery production, at a relatively high altitude (*c.* 2000 m) on the Argentera-Mercantour foothills challenges the model of early farmers’ settlement in the Liguro-Provençal arc confined to the coastal fringe. This is in line with the data obtained for the acquisition of lithic materials which demonstrate important interactions with the Alpine and Apennine worlds (Binder, De Stefanis *et al.*, this volume). In addition, this offers an important milestone for the establishment of pastoral systems based on the seasonal movement of livestock to highland pastures.

The integrated analysis, firstly naturalistic and secondly quantitative, thermodynamic and geochemical, thus opens new perspectives for the analysis of the methods of shaping and firing Neolithic pottery.

While identifying some pitfalls, this paper also demonstrates that high-resolution sourcing of materials used for ceramic production is possible and can provide a set of proxies to characterise territoriality and transfers during the establishment and evolution of early agropastoral communities.

LIST OF SUPPLEMENTARY INFORMATION AND DATA / LISTE DES INFORMATIONS ET DONNÉES SUPPLÉMENTAIRES

SD-1 – Description of the sampling contexts / *Description des contextes échantillonnés.*

SD-2 – Table of major and traces elements identified by ICP-AES and ICP-MS from geological (crude and experimentally fired) and archeological samples / *Table des éléments majeurs et des éléments-traces identifiés par ICP-AES et ICP-MS dans les échantillons géologiques de référence (crus et cuits expérimentalement) et des échantillons archéologiques.*

SD-3 – Raw chemical data and parameters for ARG45⁴⁰Ar/³⁹Ar dating / *Données chimiques brutes et paramètres de datation de l’échantillon ARG45 par la méthode ⁴⁰Ar/³⁹Ar.*

SD-4 – Examples of experimental tablets made from geological reference materials. ARG, Argentera-Mercantour massif. 1, ARG44_700°; 2, ARG33_500°; 3, ARG35_700°; 4, ARG43_900°; 5, ARG32_500° / *Exemples de tablettes expérimentales réalisées à partir des matériaux géologiques de référence. ARG, massif de l’Argentera-Mercantour. 1, ARG44_700° ; 2, ARG33_500° ; 3, ARG35_700° ; 4, ARG43_900° ; 5, ARG32_500°.*

SD-5 – Test firing for temperature monitoring at 500°C / *Test d’enregistrement des températures à 500°C.*

SD-6 – Device used for firing experimental tablets and disposition in the oven / *Dispositif utilisé pour la cuisson expérimentale des tablettes et disposition dans le four.*

SD-7 – Temperatures recorded during firing experimental tablets at 500°C / *Températures enregistrées durant la cuisson des tablettes à 500°C.*

SD-8 – Temperatures recorded during firing experimental tablets at 700°C / *Températures enregistrées durant la cuisson des tablettes à 700°C.*

SD-9 – Example of color changes of experimental tablets after firing at different temperatures of the sample ARG 32 (Argentera-Mercantour massif) / *Exemple des changements de couleur observés après cuisson des tablettes expérimentales à différentes températures pour l'échantillon ARG 32 (massif de l'Argentera-Mercantour).*

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Jean-Marc LARDEAUX

Université Côte d'Azur, CNRS, IRD, OCA,
GÉOAZUR (UMR7329)

Campus Azur

250, rue Albert Einstein
F.-06560 Valbonne

jean-marc.lardeaux@univ-cotedazur.fr

(2) Center for Lithospheric Research, Czech
Geological Survey, Klárov 3, 11821, Prague 1,
Czech Republic

<https://orcid.org/0000-0001-7666-7109>

Gilles DURRENMATH

Université Côte d'Azur, CNRS,
CEPAM (UMR7264)

MSHS Sud-Est

24, avenue des Diables Bleus
F.-06300 Nice

gilles.durrenmath@univ-cotedazur.fr

Marzia GABRIELE

Université Côte d'Azur, CNRS, IRD, OCA,
GÉOAZUR (UMR7329)

MSHS Sud-Est

24, avenue des Diables Bleus
F.-06300 Nice

marzia.gabriele@cepam.cnrs.fr

<https://orcid.org/0000-0003-0934-366X>

Chrystèle VERATI

Université Côte d'Azur, CNRS, IRD, OCA,
GÉOAZUR (UMR7329)

Campus Azur

250, rue Albert Einstein
F.-06560 Valbonne

chrystele.verati@univ-cotedazur.fr

Didier BINDER

Université Côte d'Azur, CNRS,
CEPAM (UMR7264)

MSHS Sud-Est

24, avenue des Diables Bleus

F.-06300 Nice

didier.binder@cnrs.fr

<https://orcid.org/0000-0001-8232-5367>