Physico-Chemical Study of the Neolithic Ceramics of the Region of Oran (Northwest Algeria)

Hamil Samira
Department of Human Sciences, History and Archeology, Université Center Moresli Abdellah, Tipaza, Algeria

Abstract: Neolithic sites in Oran are rich in ceramic pottery. Microscopic and chemical analysis of products is closely linked to the subsequent development of the main analytical methods: optical microscopy and X-ray fluorescence. This study learns more about the mineralogical constituents of some caves pottery shards, ceramic typology of the Oran region, likely from the basic raw material (common clay, various types of regolith, silty loams, rarely marl), knowledge of the nature of intentionally added degreasers and characterized geological and archaeological context, also combined with the microscopic study of thin pottery blades met these requirements allowing us to showcase the progress of these ancient societies.

Key words: Neolithic, pottery, prehistoric, thin blade, degreaser.

1. Introduction

Many prehistoric stations have been reported in the Oran region (Fig. 1), they refer to all stages from the Abbevillian to the Neolithic. The Neolithic age stations are characterized by the presence of pottery or polished stone. These are either open-air fireplaces located near water points, or fill deposits in naturally-dug caves.

Neolithic caves in this region are excavated in sedimentary series of carbonate nature on the southern and eastern side of Jebel Murdjadjo.

These caves were pointed out by G. Carrière in 1885 and excavated by M. Pallary and Dr Tomassini [1]. Among the remains of lithic and bone industry, there is the pottery which is known for its richness of its decoration, the originality of its style, making it a considerable element for the study of civilizations in Neolithic [2]. The Neolithic containers of the Oranais tell have a conical and wide shape with slightly narrowed openings. They often wear gripping elements, nipples, ears, whose perforation can lead to real handles. The shaping is entirely done with the pigeon.

The pasta does not translate well preparation, as for the decor, its most typical element is the incision or the groove (Fig. 2), which is reminiscent of Moroccan or Spanish ceramics [3].

Although not possessing curvilinear motifs [4], rhombuses and chevrons are commonplace, as well as punched decorations (corners, commas ...). Taking as a center of interest the elements of prehension showcase [5] real compositions.

A series of five pottery shards from different sites (Fig. 3) are:

- The forest cave located in the Mekaatel-Beydjbel on the southern slopes of the Mordjadjo massif [6].
- Grotte Ground Floor located in Chabet el Harmanne Messerghine south-west of Oran.
- Home of the Spanish Battery the station is a full-scale coastal hearth east of Oran.
- Snail-graveyard Neolithic station group. Several homes located in the dunes, at the edge of the sea on the territory of the Commune of Bou-Sfer [7], near Oran.
- Great Dar el Mandjel west of Oran.

Shards are subjected to petrographic and chemical analyses.

Corresponding author: Hamil Samira, teacher researcher, lecturer, research fields: human sciences, history and archeology.
Pulp analyses are used to determine the source of the raw materials used, to characterize the local production of a site and to identify its dissemination area. This requires a preliminary definition of this production based on quantitative and qualitative [8] criteria and on analytical results in order to find the origin of an exogenous ceramic (imported or brought), which implies the existence of a corpus of reference.

2. Materials and Methods

The study of pieces of pottery harvested from the caves studied to know more about the main components and the raw material of this product.

In general, the search for the sources of archaeological ceramics is carried out thanks to the mineralogical method which allows us to identify the
minerals present in the ceramic paste by examining the archaeological shards using a polarizing microscope. Shards stuck on a glass slide are ground and polished to a thickness of 0.3 mm uniform which allows transmission of polarized light through the polished blade [9]. In this study, we made five (5) thin blades in the laboratory of the Office of Geological and Mining Research (Boumerdes-Algeria) from the ceramic products harvested at the caves visited. The microscopic study of these thin sections was carried out at the laboratory of the Geological and Mining Research Agency (Eurl Ceramines Agency-Algiers-Algeria) using a polarizing-optical microscope of the Carl Zeiss Axiolab Pol 450910 type.

The word “dough” rather refers to raw clay, so we try to find the recipes of potters from criteria derived from the macroscopic and microscopic observation of its products.

Chemical analysis was carried out.

3. Results

The sites visited during our field trips are all located in the western part of the city of Oran with the exception of the home of the Spanish Battery east of Oran belonging to the area of the northern tell between the region from Oran to the East and the town of Targa to the West. The caves are naturally dug in the predominantly carbonate sedimentary [10] formations carried by the Jurassic lands in the Targa region and by the Miocene formations in the Masserghine region.

On the lithostratigraphic level, the Jurassic (200 to 176 Ma) of the Dhar el Mendjel massif and Sidi Kacem is represented at the base by marl conglomerate facies surmounted by marl-limestone alternations with a thickness of 20 m with levels breccia whose elements are centimetric. Sedimentation continues with massive cavernous dolomitic limestone in places with a brown patina and a gray fracture. These levels also show signs
of chemical sedimentation especially inside the cave which is marked by the formation of small concretions of stalactites and stalagmites.

Upwards, the series is marked by crystalline limestones from black patina to bluish with a gray black break. This formation is affected by fractures of different sizes, which are mostly filled by calcite. This level shows on its surface a pronounced brecciation where one can find elements composed essentially by these crystalline limestones taken up in a carbonate cement.

The Miocene (23 to 5 Ma) is observed in the Messerghine region [11] where it is materialized at the base by marly-calcareous yellow levels more or less friable with a thickness estimated at 10 m. They are surmounted by sandstone limestones with dendrites of manganese. This level which often shelters the caves presents a thickness of 8 m. The whole is capped by white-beige massive limestones, which show a direction 20° N, 20° E.

During the comparative analyses of the samples, the mineral and petrographic composition, the particle size and the texture of the ceramic mass were taken into consideration.

The projected approach on all the ceramic products of the different sites studied based on the observation of the thin sections enabled us to translate and characterize the nature of the elements which constitute the pasta studied in each site. The thin blade study of the prehistoric ceramic products of Oranais shows that they used various recipes to make their ceramics [12], in detail the points raised are as follows.

3.1 Cave of the Forest

Macroscopically, the studied remains present very heterogeneous pastas or ceramic fabrics [13], with massive, crystalline and grainy textures. The microscopic study revealed that it is coarse facies with phenocrysts of quartz and feldspars of the orthoclase, microcline and albite type cemented by a very dark

Fig. 4  Color photomicrographs of thin sections of five pieces of ceramics. 10× magnification, polarized light cave of the forest.
haematitized pelagic clay paste. The lithic elements present are of variable size, which does not exceed the 4 mm of diameter classified like rudite (2 mm) according to the scale of Wentworth [14]. They are polygenic whose nature is silty to pelitic and rarely carbonated. Their shape is rounded to sub rounded. There are also some traces of spherical bioclasts and opaque minerals (Fig. 4). The degreaser consists of small angular quartz grains, some fragments of shells and charcoal or recycled chamotte [6].

3.2 Ground Floor

It is a very fine clay to manganese carbonate facies of blackish color. The ceramic paste is very fine consisting of clay minerals (75%) containing lithic elements (10%) of 1 to 3 mm in diameter included in the class of rudite arenites [14] of detrital detritic nature, clayey (Fig. 5), ferriferous and rarely silty and carbonated. These elements are composed of grains often rounded with a fine cement and are generally surrounded by a carbonate halo and the whole is crossed by a network of micro-fractures filled with calcite and sometimes secondary silica.

3.3 Spanish Battery (Hearth the First Sheepfold)

The ceramic fabric has a lamination with a certain preferential orientation of the elements. This paste is fine and composed of clay and ferro-manganese (Fig. 6) minerals which give it the color red to black. It contains many more sub-round quartz grains that result from several transport cycles and could have come from alluvial deposits, the beach or coastal dunes. Added to this is some rare orthoclase plagioclase in very small grains. The calcite is either in isolated grains, it is filled with micro-fractures or it envelops the lithic elements that bathe in this paste. A small percentage (1%) of isolated opaque grain minerals is noted.

![Color photomicrographs of thin sections of five pieces of ceramics. 10× magnification, polarized light ground floor.](image)
The lithic components are less abundant (10%) than the studied samples and they are oxidized clay (hematitized and pelagic).

3.4 Escargots Cemetery

The microscopic study of this thin plate shows a fine clayey to sericitous facies very poor in lithic elements, which present only 5% of the total volume and with a spherical shape, a size of 1 mm in diameter considered as arenite according to Ref. [14], a red color and a fine and homogeneous particle size. A rate of 5% of quartz is observed as an abundant element (Fig. 7).

3.5 Dar El Mandjel Cave

The microscopic study of this sample revealed that the paste is clayey, fine, ferruginized, hematitized and red in color. The clay minerals constitute a volume of 60% to 70%. We note the existence of millimetric-sized quartz grains and calcite of xenomorphic to sub automorphic form with rare ortho-type plagioclases marked by polysynthetic macula-invading twins at the level of cleavages. Chalcedony grains are also identified in this thin blade in the form of fibro-radiate minerals. The lithic fragments are very abundant (30%-40%) and are cemented by the clay fraction. They have a size that does not exceed 3 mm in diameter, the nature of its elements is heterogeneous: clay, shale, pelitous, carbonated and sometimes silty, opaque (Fig. 8) minerals are not present, the prehistoric potter deliberately added during shaping.

Fig. 6  Color photomicrographs of thin sections of five pieces of ceramics. 10× magnification, polarized light.
4. Chemical Analyses

The equipment used is of the “energy dispersive X rays fluorescence” type, ED 2000 brand of the nuclear physics laboratory of Algiers. The X-ray tube is composed of a silver anticathode and the detector of a Si semiconductor cooled by liquid nitrogen. This type of instrument allows the characterization and quantification of chemical elements (Table 1).

Through this approach, we wish to highlight the information potential that sediments contain precisely and integrate both environmental changes and the impacts of human activities on the natural environment.

5. Interpretation of Chemical Analyses

5.1 Ground Floor Cave

This sample shows a high level of silicon (Si = 23.51%) and oxygen (O = 54.19%), which indicates the presence of a high silica (\(\text{SiO}_2\)) content represented by the sand which has been used as a degreaser in this Neolithic age pottery formula. It is necessary to point out the considerable level of alumina (Al = 8.4%) resulting from the clay fraction also used in the ceramic recipe (Fig. 9).

The presence of potassium is also significant and testifies the existence of orthosis (potassium feldspar) as one of the components of this recipe. The brown color of these pottery fragments is only the influence of the iron oxides with a rate equal to 2.34%.

5.2 Cave of the Forest

The chemical X-ray fluorescence analysis of this sample reveals almost the same chemical characteristics as ground floor pottery with a silicon content exceeding 23%. This element combines with a part of oxygen to form silica grains (main component of sand degreaser). Alumina is present in this amalgam
Color photomicrographs of thin sections of five pieces of ceramics. 10× magnification, polarized light Dar El Mandjel Cave.

| Elements | Rez de chaussé | Cave of the Forest | Spanish Battery | Dhar El Manjdel Cave | Ain Gueddara Cave |
|----------|----------------|--------------------|-----------------|---------------------|------------------|
| C        | 4.548          | 3.841              | 5.377           | 4.134               | 4.134            |
| O        | 54.129         | 54.835             | 49.952          | 49.72               | 49.472           |
| Na       | 1.248          | 0.995              | 0.927           | 0.781               | 0.781            |
| Mg       | 1.042          | 1.444              | 1.410           | 8.005               | 1.498            |
| Al       | 8.486          | 8.510              | 8.662           | 20.054              | 9.478            |
| Si       | 23.512         | 23.333             | 20.706          | 0.364               | 27.854           |
| P        | 0.097          | 0.172              | 0.150           | 0.107               | 0.189            |
| Cl       | 0.063          | 0.118              | 0.058           | 0.055               | 0.050            |
| K        | 2.657          | 2.452              | 0.439           | 3.168               | 0.553            |
| Ca       | 1.542          | 1.320              | 1.698           | 8.314               | 2.973            |
| Ti       | 0.226          | 0.197              | 5.712           | 0.541               | 1.698            |
| Mn       | 0.027          | 2.685              | 0.431           | 0.058               | 0.300            |
| Fe       | 2.344          | 0.011              | 0.025           | 4.752               | 0.042            |
| Zn       | 0.003          | 0.013              | 4.307           | 0.013               | 4.335            |
| Rb       | 0.009          | 0.013              | 0.014           | 0.008               | 0.014            |
| Sr       | 0.029          | 0.002              | 0.008           | 0.029               | 0.007            |
| Y        | 0.004          | 0.025              | 0.004           | 0.063               | 0.003            |
| Nb       | 0.002          | 0.027              | 0.058           | 0.025               | 0.019            |
| Ba       | 0.031          | 0.006              | 0.002           | 0.055               | 0.021            |
| Zr       | 0.023          | 0.002              | 4.134           | 0.008               | 0.008            |
| W        | 0.025          | O                  | 49.472          | 0.037               | 0.008            |
| Pb       | 0.012          | Na                 | 0.781           | Tc                  | 0.008            |
with the same rate as that of the first sample, which forms a good part of the clay phase. The presence of K and Ca is probably due to the presence of feldspars.

The oxide used for the coloring is the manganese oxide, this is interpreted by the remarkable rate of Mn = 2.68%, which explains the dark (black) color of this sample (moreover it is the only sample where the Mn level is abnormal).

5.3 Spanish Battery

This sample shows a lower Si content than the other two previous samples and therefore a decrease in the use of degreasing sand. This deficit is argued by the marly and clay nature of the fine component where we note an enrichment in terms of calcium compared to the first two (Ca: 5.71%) in addition to a remarkable decrease of potassium K: 1.69% which also indicates the decrease of the ortho grains in this sample.

It should be noted that the appreciable level of Na and Cl and even the presence of sulfur and Sr can indicate a salinity level within the materials used.

As regards the coloring used, the use of iron oxide (Fe: 4.3%) in contrast to the previous sample is noted.

5.4 Cave of Dar el Mendjel

This fragment of pottery indicates the use of a marly component clay is important which is due to a significant and important presence of Ca: 8.31% (it is the most important rate among all the samples) which composes with coal and calcium carbonate (known as calcite whose chemical formula is CaCO₃).

The rate of silica (sand grain) is lower compared to the first two samples and is close to that of the Spanish Battery sample (Si: 20%).

Orthoclase is expressed by the presence of potassium (K: 3.16%) and also the clay fraction by the presence of aluminum (Al: 8.00%).

The coloring in this case is ensured by the presence of 4.75% iron oxides.

5.5 The Cave of Ain Gueddara

This sample shows the highest rate of degreasing sand (Si: 27.85%) and also the importance of the clay fraction (Al: 9.47%) and the decrease of calcium carbonates (Ca: 1.69%). Potassium feldspar is well expressed in this fragment (K: 2.97%), the coloration is ensured by the presence of Fe oxide: 4.335.

6. Conclusion

The microscopic study of shards shows the use of several raw materials in the ceramic production recipe such as clays, lithic fragments of different types and iron oxides (hematites) and also manganese oxides. This approach has shown us that there are two pottery-making recipes in Oranea.

The first family includes samples made from quartz that will be derived from coastal or alluvial sand as a degreaser especially in the areas near the coast as in the case of the Cave of the Spanish Battery and that of Cemetery Snails that contain a considerable rate of rounded and blunted quartz and a low level of lithic elements. These are mixed with clay and do not contain a mixture with crushed rock because they show a limited amount of lithic fragments.

The presence of the remains of an old chamotte indicates the recycling of the latter especially in the shard of the cave of Dhar El Mendjel.

In the second family groups the samples composed mainly of silty clay and a limited rate of quartz and with a considerable rate of lithic elements (silt, pelite, shale, carbonate) as in the case of the cave of Dar el Mendjel, the cave of the Forest and that of ground floor where they constitute a proportion between 30% and 40%. These rock fragments could come from sediments associated with clay (main raw material) or intentionally added to the recipe during the manufacture of ceramic products.

The studied samples are distinguished by their rather homogeneous character having common mineralogical characteristics and the homogeneity of the paste and
the degreaser of the ceramic is explained by the geological situation. On this basis it can be concluded that the Miocene clay from the vicinity of the studied caves was used in pottery ceramics as one of the raw materials. This argument is based on the geological potential of the clay in the basins that surround the formations sheltering the caves in question.

In raw material supply strategies, there are two types of ceramics, one made from local raw material at the site of the Spanish Battery and that of the Snail Cemetery which are just a few meters from the rich beach. In quartz and the other type the supply of clay was made in a territory of a few kilometers or comes from the surroundings in plains rich in silty clay.

The majority of the samples follow the same trend in terms of chemical elements, except some differentiations due to the origin of the material used in the manufacturing formula of this pottery.

According to these results, we can obtain the following observations:

- The silica (sand grain) content is important, especially in Ain Guedara shards (27%). In general, the Si content is greater than 20%. There is a relationship between the contents of Si and Ca (that is, when Ca is moderate the Si is dominant and vice versa).
- For Al, it shows an average rate of 8%.
- The coloring is generally carried out by iron oxide except in the case of the sample of the cave of the forest.

According to these analytical data, it was possible to follow the evolution of the major elements (i.e.) C, O, Na Mg, Al Si, P, Cl, K, Ca, Ti, Mn, and Fe, as a function of samples collected in the field as a binary graph in Fig. 9.

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