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Reading ceramic surfaces:

² characterisation of surface treatments

₃ towards functional identification of vases.

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12 Abstract

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- 13 This study explores the characterisation of clay vases surfaces through tribological analysis
- and more precisely the relationship between surface treatments and watertightness. Different
- surface treatments were tested (pre-firing: smoothing with a wet hand, a wooden tool, a piece
- of cloth, burnishing using a pebble and a combination of a pebble and a piece of leather; and
- post-firing organic coatings: milk and fleece on red hot surfaces and olive and beeswax applied
- to the room temperature clay tablets). Surfaces were measured and characterised using a
- 19 confocal rugosimeter, as well as the method of continuous wavelet transform. These multiscale
- 20 analyses allowed the classification of surface treatments into four morphological groups.
- 21 Furthermore, water and oil drop deposition tests allowed the characterisation of a surface's
- reaction when exposed to liquid contents. These results have implications in evaluating
- storage and cooking performances and specific uses of clay vases in relation with organic
- materials. The discussion will also provide insights regarding the importance of the first use of
- a pottery in the *chaîne opératoire* of manufacture.

Introduction

- 27 As regards the function of clay vases, morphological, technological and functional
- studies are used to investigate the potter's choices and their influence on vessels'
- 29 performance characteristics. Further discussions have showcased the extent to which
- these characteristics are adapted to vases' intended function (inter alia Bonaventure,
- 2010; De Ceuninck, 1994; Regert et al., 1998; Tsirtsoni and Vouni, 2000; Vieugué et
- 32 al., 2008). In this framework, experiments in controlled laboratory conditions have
- multiplied, during the late 1980s and early 1990s (Bronitsky and Hamer, 1986; Schiffer
- 34 *et al.*, 1994; Skibo, 1990; Young and Stone, 1990).
- From the raw material to the surface treatments, the *chaîne opératoire* of ceramics
- manufacturing entails a series of actions and parameters that produce a usable vessel
- 37 (Soressi and Geneste 2011 pp. 334-350). Given the complexity of various parameters,
- 38 functional studies have focused on specific performance characteristics, such as
- impact resistance, tensile strength, abrasion resistance, portability, thermal resistance

- 40 and heat distribution efficiency, watertightness etc. (Braun, 1983; Bronitsky and
- 41 Hamer, 1986; Drieu, 2017; Hein et al., 2015, 2009; Lepère, 2009; Müller et al., 2014;
- 42 Rageot, 2017; Rice, 1987; Rye, 1981; Schiffer et al., 1994; Schiffer and Skibo, 1987;
- 43 Skibo, 1990, 1992; Steponaitis, 1984; Tite *et al.*, 2001; Young and Stone, 1990).
- 44 Functional approaches offer insights into what a pottery was made for, however, not
- on how it was effectively used during its "life", the study of which, has been conducted
- by use-wear and residue analysis (Charters et al., 1993; Condamin et al., 1976; Drieu,
- 2017; Evershed *et al.*, 1997; Evershed *et al.*, 2002; Fanti, 2015; Formenti, 1989; Forte
- 48 et al., 2018; Griffiths, 1978; Regert et al., 1999; Skibo, 1992, 2012; Vieugué, 2014).
- 49
- As part of our broader study on Prehistoric ceramics in the Mediterranean area (South
- of France [3200-2300 B.C.] and Crete [3200 1100 B.C.]), we have investigated the
- role of surface treatments on the function of clay vases.
- 53 Surface treatments even out the surface and push the temper below it (Shepard, 1956).
- 54 They have long been considered from an aesthetic standpoint. Nonetheless, by
- modifying the surface microtopography and suppressing micro-porosities, the potter's
- technical actions also alter the vessels technical properties. Among various surface
- 57 treatments that have been identified, smoothing, burnishing and polishing can be
- considered the most common (Balfet, 1966; Lepère, 2009; Lepère, 2014; Martineau,
- 59 2010; Rice, 1987; Rye, 1981; Shepard, 1956). Ethnoarchaeological investigations and
- experiments have shown that such techniques differ depending on the type of clay,
- 61 "technical gestures", strength exerted, tools and, overall, hygrometry of the fabric
- 62 (Balfet et al., 1989; Lepère, 2009; Martineau, 2010; Rice, 1987; Roux and Courty-
- Fedoroff, 2017; Rye, 1981; Shepard, 1956). Based on visual and tactile criteria, such
- studies have helped identify technological characteristics of surface treatments at a
- 65 macroscopic scale.
- 66 However, many surface treatments using organic materials remain understudied, due
- to conservation bias. The main function of organic coatings is to slow water
- 68 permeability and to bring content to a boil. Indeed, low fired pots without any
- waterproofing coating cannot reach boiling temperature. However, it should be noted
- that the goal is not always to reach a boil but can be to simmer the content. To achieve
- this, coating is not necessary (Skibo, 1992). Their identification in the archaeological
- 72 record can be achieved under certain condition, using gas chromatography/mass
- spectrometry analysis (Drieu, 2017). These types of studies are rarely applied in the
- 74 archaeological record although ethnographic investigations regularly highlight the
- omnipresence of organic materials in pottery surface treatments (Gosselain, 2002;
- Livingstone Smith, 2010; Schiffer, 1990). Moreover, distinguishing organic coatings on
- archaeological vases from organic deposits related to the vases' function remains a
- 78 challenge.
- The establishment of a reference database of surface treatments applied to flat clay
- tablets, aims firstly at identifying signatures of organic and mineral surface treatments
- at a microscopic scale; secondly, characterising surfaces' performance characteristics

depending on their treatments. Tribological analysis was applied in order to identify specific signatures and performances (in this case their hydrophobic nature).

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1. Methodology

1.1. Insights on various surface treatments in the prehistoric Mediterranean

- Pre-firing surface treatments such as smoothing, burnishing and slip coating, regularly
- identified in the archaeological record, were applied with tools available during the
- Neolithic and the Bronze Age in the South of France and Greece, namely a hard
- 90 pebble, a wooden smoother, a piece of leather, a piece of cloth and bare hands
- 91 (Lepère, 2014; Martineau, 2010; Vieugué et al., 2008).
- The organic materials we used for our experiments are all attested by residue analysis.
- Beeswax (Decavallas, 2011; Regert et al., 1998), olive oil (among others Beck et al.,
- 2008; Condamin *et al.*, 1976; Copley *et al.*, 2005), and milk (Drieu, 2017) have been
- 95 identified in ceramic containers and were readily available resources in the Prehistoric
- 96 Mediterranean. Residue analysis cannot yet clarify whether these substances were
- 97 part of the diet, the surface treatment or both. Other possible organic surface
- treatments, of plant and animal origin, will be investigated in a broader study (see
- 99 discussion).

1.2. Experimental material

- 101 12 tablets, 5 square centimetres wide and 1.5 centimetres thick, were made for the
- purposes of this experiment. We used raw materials found in karstic areas around the
- Mediterranean: natural clay, sourced from Saugras (Hérault, France) and carbonate
- temper (calcite and aragonite) from a cave in Saint-Pons-de-Thomières (Hérault
- 105 France).
- After being cleared of its impurities, the clay was sorted. The temper was crushed and
- sieved, to retain only grains thinner than 1 millimetre. Eventually, the temper was
- added to the extent of 25% of the clay mass. The tablets were fired at 750°C for four
- hours in an electric kiln, and thus under a controlled atmosphere. The temperature was
- defined according to different works on open-firing temperatures (Shepard 1956: 84;
- 111 Martineau and Pétrequin 2000: 345). These sources have shown that 750°C is
- coherent for prehistoric firings.

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- The surface treatments carried out for this experiment can be classified into two
- categories depending on whether they were applied pre-firing or post-firing (figure 1,
- table 1). Six tablets fall under the first category. Smoothing was either applied with a
- wet hand (1), a piece of cloth (2) or a wooden smoother (3). When applied, burnishing
- was conducted about 40 minutes after smoothing with a smooth pebble (4) and, in one
- case, after being dried, rubbed with a piece of leather (a. k. a. furbishing) (5). One
- tablet was slip coated after being smoothed and dried (8).

One other tablet was smoothed using a wooden smoother and six others burnished with a smooth pebble before firing. After being fired, they were either coated with olive oil (6), beeswax (13S and 13B), fleece (sheep wool) (7) and milk (10S and 10B). Olive oil was simply added on the surface after cooling, much like a pottery would naturally absorb during the storing of olive oil. Beeswax was melted and applied to the surface of tablets warmed beforehand. Experimental data have shown that this technique is the most efficient (Debels 2019). Finally, milk and fleece were added onto red hot surfaces (around 540 °C). Regarding technical actions, olive oil and milk were dipped, beeswax was smeared and fleece was dabbed. Milk and beeswax smearing were tested both on a smoothed and a burnished surface for comparative purposes.

Pre-firing techniques

Post-firing techniques



Figure 1: The experimental tablets.

N°	Tools	Prefiring treatment	Post-firing treatment	Technical gesture (post-firing)	Group
1	Wet hand	Smoothing	Ø		I
2	Cloth	Smoothing	Ø		I
3	Wood	Smoothing	Ø		I
8	Slip coating	Smoothing	Ø		П
4	Pebble	Burnishing	Ø		Ш
5	Pebble, leather	Burnishing	Ø		Ш
6	Pebble	Burnishing	Olive oil	Dipping	IV
7	Pebble	Burnishing	Fleece on red hot surface	Dabbing	IV
10S	Wood	Burnishing	Milk on red hot surface	Dipping	Ш
10B	Pebble	Burnishing	Milk on red hot surface	Dipping	IV
135	Pebble	Smoothing	Beeswax	Smearing	П
13B	Pebble	Burnishing	Beeswax	Smearing	IV

Table 1: Finishing techniques, tools, pre- and post-firing treatments used in the experiment.

1.3. Macro-scale surface measurement with a confocal rugosimeter

Measurements were conducted, following a proven methodology applied to lithic 136 artefacts (Anderson et al., 2006; Astruc et al., 2003; Boleti et al., 2016; Morero et al., 137 2013; Procopiou et al., 2011, 2013; Vargiolu et al., 2007). We measured surface 138 topography with a confocal rugosimeter. This optical device uses chromatic aberration 139 which focuses different wavelengths of white light at different points. These focused 140 wavelengths pass through a filter while others are removed. An associated confocal 141 microscope selects the focused wavelengths and analyses with a spectrometer the 142 wavelength with the highest intensity. Knowledge of the relationship between the 143 144 wavelength and the distance of the focal point allows the measurement of roughness. For our case study the area of the surface measured was 25 square mm (1/10th of the 145 tablet area) with a resolution of 2 µm. 146

1.4. Multi-scale analysis: the method of continuous wavelet transform

- This multi-scale analysis characterises the morphology of striations and the micro roughness of polish. Observation of these marks on stone vases and tools has for example been used to identify identify the abrasives employed as well as lubricants and their viscosity (Procopiou *et al.* 2013; Boleti *et al.*, 2016).
- As the Newton's prism decomposes white light into its spectral components, the wavelet transform decomposes a topographic signal into its different scales: roughness, undulation, shape. This multi-scale decomposition allows to quantify the decomposed scales with the roughness coefficient SMa, which reflects the arithmetic mean value of the signal's amplitude for a given wavelength. The results is presented as a SMa spectrum, which constitutes a multi-scale signature of the topographic signal of the surface (figure 2).
- A wide range of wavelengths and amplitudes: z(x, y) = f(Li, Lj) are generally contained in the distribution of the heights of a surface, corresponding to the three-dimensional signal z(x, y). Classifying wavelengths of roughness and waviness of a solid conventionally depends on wavelength bands: shape (flat surface, cylinder, sphere: $L > 2500 \mu m$), waviness ($L < 2500 \mu m$), microscopic roughness ($L < 2500 \mu m$) and nanometric roughness (a few nm < L).
- The analysis of the geometrical aspects of the surface can be achieved using three scales of observation: shape, waviness and roughness. The first and second of these, permit the identification of manufacturing techniques. The third, allows the identification of polishing techniques (Procopiou, *et al.*, 2011: 2528).
- "The spectrum of multi-scale decomposition of a surface is obtained by the coefficient SMa [...] representing the arithmetic mean value of all the scales of the surface expressed as follows" (Lee 1997; Lee *et al.* 1998; Mezghani 2005; Zahouani & El Mansori 2017).
- 173 In our case, we present the average wavelengths of three different measurements on each surface.

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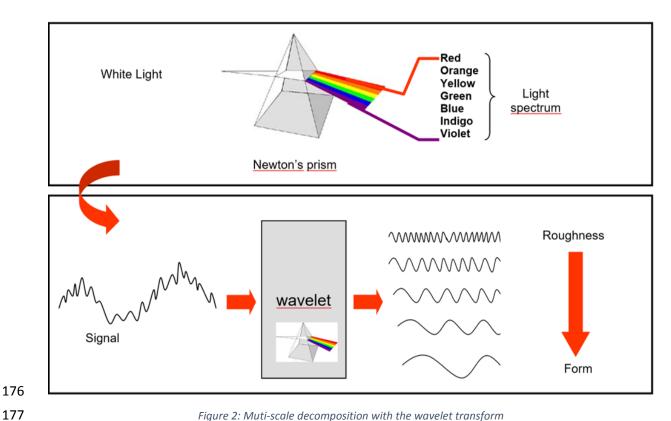


Figure 2: Muti-scale decomposition with the wavelet transform

1.5. Needle-free drop deposition technique for contact angle

We also used drop shape analysis to evaluate the wettability of the surfaces, according to the surface treatment (figure 3). This apparatus uses a syringe that deposes a drop of a liquid substance on a plane surface (Stanimirova et al., 2019). The duration of the deposition as well as the evolution of the angles formed between the contour of the drop and the flat surface are measured with an optical device and filmed by a camera opposite a light source.

We first deposited a 0.050 ml drop of water. The experiment was then repeated with a drop of oil of the same amount of liquid. Measurements were conducted 5, 10 and 15 seconds after drop deposition.

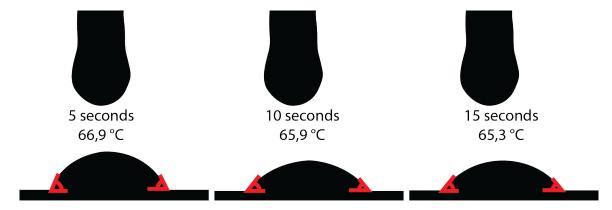


Figure 3: Contact angle measurement with the needle-free drop deposition technique.

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190 2. Results

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2.1. Surface measurement and characterisation

- Four groups of topographies can be observed (figure 4). The first two correspond to the smoothed surfaces and the last two to the burnished surfaces. Surfaces in groups
- the smoothed surfaces and the last two to the burnished surfaces. Surfaces in groups I and III are plain, while surfaces in groups II and IV are coated. Our description of
- states of surfaces will follow the terminology already defined by several scholars (for
- example Buckley 1981).
- 197 It should be noted that no data could be recorded for tablets 7 and 10B, as their surface
- was too dark or too white to be measured.
- Surfaces in group I present an uneven topography. Striations are deep, unidirectional,
- linear with a v-shaped cross section, strongly closed to strongly connected. Edges
- 201 have an irregular morphology. The coarse texture results from the uneven spreading
- of clay.
- Surfaces in group II also present an uneven topography, though slightly more regular.
- Striations are less deep. They are unidirectional, linear, with a V-shaped cross section
- and strongly connected. Edges have an irregular morphology. The coarse texture
- results from the uneven spreading of clay. Coating reduces surface roughness by
- 207 decreasing asperities.
- Surfaces in group III are flat to sinuous. They are smooth, closed, shallow, with
- 209 rounded to sharp edges. The flat texture results from a homogeneous spreading of
- 210 clay.
- Surfaces in group IV are flat, smooth, closed, shallow, with rounded edges. The flat
- 212 texture results from a homogeneous spreading of clay, thus coating the asperities.

2.2. Surface characterisation with the method of the continuous wavelet transform

- 215 The method of the continuous wavelet transform shows distinct signatures for
- smoothed/burnished surfaces (between 0.00024 and 0.244 mm) and polished ones
- 217 (between 0.0625 and 1 mm). Smoothing/burnishing thus produces a larger scale of
- wavelengths than polishing while the addition of coating reduces the gap (figure 5).
- 219 These results therefore confirm the relevance of macroscopic characterization of
- surfaces and provide, for the first time, roughness signatures of clay archaeological
- surfaces depending on their type and degree of treatment.

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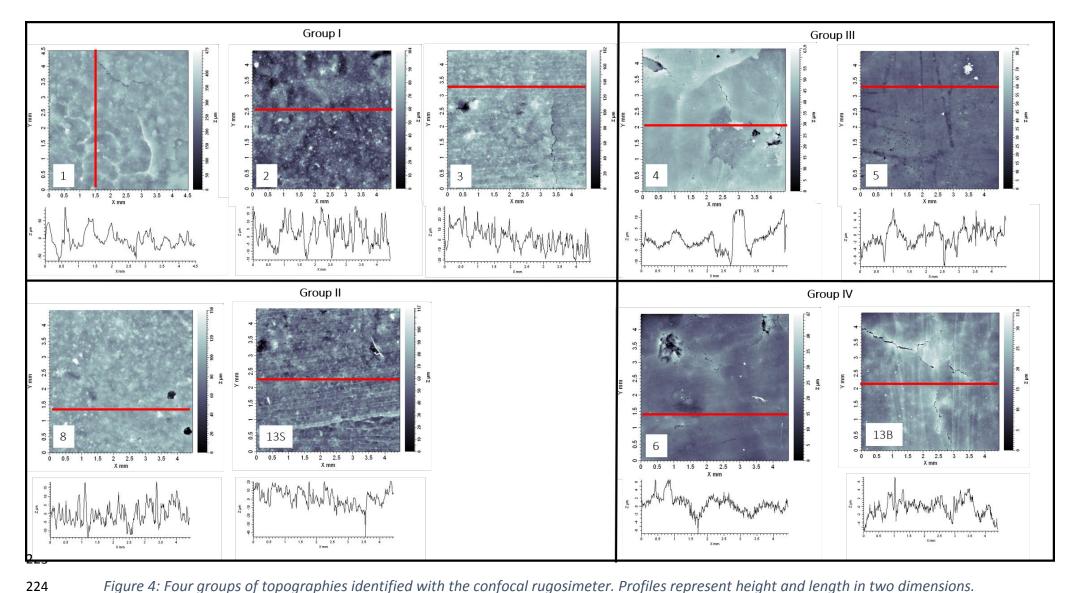


Figure 4: Four groups of topographies identified with the confocal rugosimeter. Profiles represent height and length in two dimensions.

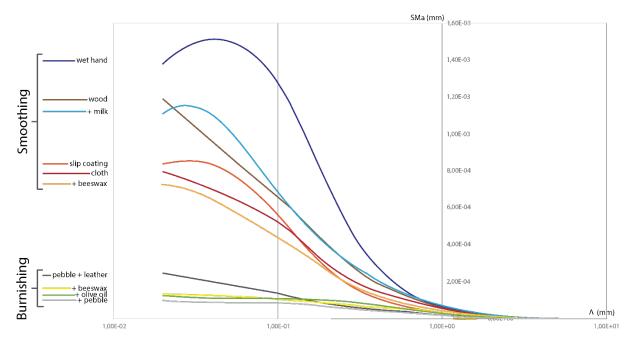


Figure 5: Burnishing and smoothing: two distinct signatures identified with the method of continuous wavelet.

2.3. Surfaces' behaviours towards water drop deposition

Water drop deposition showed that post-firing surface treatments have a clear sealing effect (figure 6), whatever the underlying surface, as had already been noticed by other scholars (Drieu, 2017; Gosselain, 2002; Lepère, 2014; Longacre et al., 2000; Schiffer, 1990). It should be noted that no data could be recorded for tablet 7, as its porosity directly absorbed the drops.

Angles of water drops were highest when deposited on surfaces coated with beeswax, milk and olive oil: ranging from 50 to 70°, they stood still over the span of 15 seconds and then held for at least 100 seconds.

On the contrary, angles of water drops deposited on plain surfaces, whether smoothed or burnished, appeared to be significantly lower. Ranging from 0 to 30°, they decreased quickly during the span of 15 seconds, though, the drops on the burnished tablets 4 and 5 were steadier.

On average, the water drops deposited on the post-fired treated surface evolved to lose 1.26° while the pre-fired treated surfaces lost 9.7° on average, over 15 seconds, hinting that the latter might completely absorb water contents that is then subject to evaporation through pores over time. Je fais appel à ma reference.

In other words, post-firing treatments increase hydrophobic properties of a surface which, otherwise, would remain relatively hydrophilic.

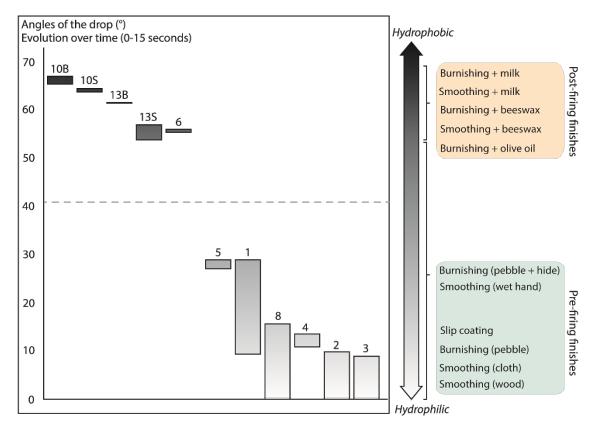


Figure 6: Results of the water drop deposition on the experimental tablets. The rectangles' length represents the evolution over a 15 second span

2.4. Surfaces' behaviours towards oil drop deposition

This experiment was carried out in order to evaluate the behaviour of a surface towards a fatty product, in comparison with water. Indeed, it is very well-known that, since the Neolithic, many pots in the archaeological record were used to store, cook or pour fat products, among which vegetable oils. For the purposes of this experiment, olive oil was chosen, since its use started to spread around the Mediterranean during the Bronze Age.

With the exception of tablet 6, post-firing surface treatments, along with slip coating, proved once again to have the best sealing properties, whatever the underlying surface (figure 7). Ranging from 63.2 to 36.5°, the angles ranged from 43.2 to 24.9° after 15 seconds.

Oil drops' angles on pre-firing surface treatments presented systematically lower values. Ranging from 31.1 to 19.9°, they ranged from 25 to 18° after 15 seconds.

In other words, post-firing treatments increase lipophobic properties of a surface which, otherwise, would remain quite lipophilic.

Despite the similarities with the water drop behaviour, one can notice that the angles' average values decrease quickly, especially in the case of surfaces treated before firing. These preliminary results suggest that the behaviour of the drop depends upon

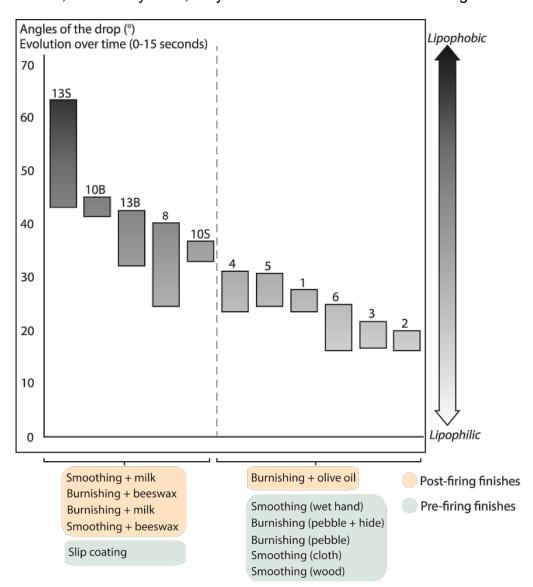


Figure 7: Results of the oil drop deposition on the experimental tablets. Rectangle length represents evolution over 15 seconds

3. Discussion: Surface treatments and performance characteristics of clay vases

These preliminary results show the influence of surface treatments on the waterproofing of clay vases. Based on tribological methods, we measured the roughness of clay tablets at a microscopic scale and the contact angle of water and oil drops deposited on these surfaces. A wide range of surface treatments, based on archaeological, experimental and ethnographic observations, were tested but the

reference database still needs enlarging, especially with regard to organic materials.

We have so far focused on milk, wool, olive oil and beeswax but other materials such

as animal fats, birch bark tar, etc. should be considered in the future.

288 Roughness was measured with a confocal microscope and characterised by the

method of the continuous wavelet transform, providing, for the first time, a microscopic

signature of surface treatments. These reference data must be compared, in the future,

with those from the archaeological record.

In fact, ceramic surface attrition constitutes one of the major research areas in the field of surface characterisation. Although well developed by a small number of scholars (Bonaventure, 2010; Fanti, 2015; Forte *et al.*, 2018; Saurel, 2017; Skibo, 1992; Vieugué, 2014), the use-wear approach could be strengthened by borrowing methodologies specific to tribology. The precise characterisation of surfaces undertaken in this paper constitutes the first step towards a better understanding of the state of surfaces depending on the surface treatments. The works of J. M. Skibo, T. C. Butts and M. B. Schiffer have already shown that abrasion resistance is directly influenced by the firing temperature and the nature of the surface (Skibo *et al.*, 1997).

Neither chemistry nor microscopic analysis can yet differentiate the initial motivation behind the formation of organic residues, that is to say determine whether they reflect an intentional coating or the content of the vase. However, prehistoric users may not have differentiated between the two stages. Indeed, the first uses of a cooking pot can aim at testing the integrity of the vessel, at removing or lessening the earthy taste and waterproofing the container. Moreover, some studies suggest that boiling organic material in a vase before use will not only waterproof it but will also significantly reduce the time needed to reach boiling temperatures in the future (R. P. Evershed et al., 1997; Skibo, 1992). By taking this into perspective, the first use and the last stage of the *chaîne opératoire*, the organic coating, can in some case be superimposed (figure 8). A number of coatings can be obtained by applying organic material on a warm pot or simply by warming it up inside the vessel (Gosselain, 2002; Harry *et al.*, 2009; Henrickson and McDonald, 1983; Rye, 1981). In the case of edibles, the content can potentially be consumed afterwards.

Furthermore, experimental data have shown that the first use of cooking pots will not completely saturate its clay matrix with lipids, which continue to accumulate throughout its lifetime (Debono Spiteri, 2012; Evershed *et al.*, 1997). Successive cooking sessions will participate in the reinforcement of the coatings, and thus, become a part of the "maintenance stage". Only an intensive first use (with a significant amount of lipids over a long period etc.) can potentially saturate the clay matrix. However, successive uses of subtractive nature (such as abrasions and erosions) will eventually thin out the coating which will require further maintenance.

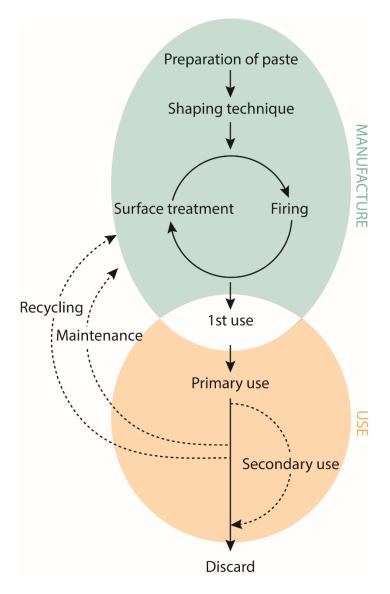


Figure 8: Flow model of pottery manufacture and use (after Schiffer, 1972, p. 159).

This study opens new perspectives for the functional analysis of ancient clay vases, and the reconstruction of past practices. Through macro- and microscopic observations as well as residue analysis, pre-firing surface treatments and post-firing coatings can be characterised. This contributes to inform on potters' choices and users' actions aimed at maintaining the expected performance of the pot.

Characterising the behaviour of surfaces towards water and oil drops can contribute strongly, among other further developments, to evaluating storage and cooking performances and the specific uses of clay vases in relation with organic materials. Concerning storage, for instance, we have suggested that these organic materials, whether reflecting surface treatment or contents, could reinforce the hydro- and lipophobic properties of vases. They thus increase storage performances and, as far as possible, should be taken into account along with vases' capacities in order to improve storage estimations.

Lamps, which in the Mediterranean area mainly used animal fats, beeswax or vegetable oil the from Neolithic onwards (Charters et al., 1995; Condamin *et al.*, 1976; Copley *et al.*, 2005; Evershed *et al.*, 1997; Evershed *et al.*, 2002; Garnier, 2002; Garnier *et al.*, 2011, 2009; Mayyas *et al.*, 2017; Tsirtsoni and Bourguignon, 2016) offer a relevant case study, as the combustion duration depends not only on their capacity but also on the fuels used (Rueff, 2019). Combustion duration is, for instance, 5.5 times longer with olive oil than with beeswax in a smoothed lamp. An experimental burnished lamp having a capacity of 0.25 I of olive oil, reached 41 hours and 58 minutes without any kind of maintenance - adjusting the wick for instance. Experiments conducted with the tablets suggest that in a smoothed lamp with the same capacity, the burning length would be 1.3 times shorter than in the burnished one. We can estimate, for instance, a duration of 32 hours and 28 minutes (2518 / 1.3 / 60).

More than just aesthetic features, surface treatments have a clear function in the use of pottery, by improving its waterproofness and increasing its performance. By extending this tribological approach in the future and by combining it with chemical residue analysis, we aim to define, diagnostic criteria of finishing techniques and modes of pottery use.

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 Wear
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