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Bastien Rueff, Pauline Debels, Roberto Vargiolu, Hassan Zahouani, Haris Procopiou. Reading ceramic surfaces: Characterisation of surface treatments towards functional identification of vases. *Journal of Archaeological Science: Reports*, 2021, 38, pp.103021. 10.1016/j.jasrep.2021.103021 . hal-03468734

HAL Id: hal-03468734

<https://hal.science/hal-03468734>

Submitted on 13 Jun 2023

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1 Reading ceramic surfaces: 2 characterisation of surface treatments 3 towards functional identification of vases. 4

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11

12 Abstract

13 This study explores the characterisation of clay vases surfaces through tribological analysis
14 and more precisely the relationship between surface treatments and watertightness. Different
15 surface treatments were tested (pre-firing: smoothing with a wet hand, a wooden tool, a piece
16 of cloth, burnishing using a pebble and a combination of a pebble and a piece of leather; and
17 post-firing organic coatings: milk and fleece on red hot surfaces and olive and beeswax applied
18 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
22 reaction when exposed to liquid contents. These results have implications in evaluating
23 storage and cooking performances and specific uses of clay vases in relation with organic
24 materials. The discussion will also provide insights regarding the importance of the first use of
25 a pottery in the *chaîne opératoire* of manufacture.

26 Introduction

27 As regards the function of clay vases, morphological, technological and functional
28 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
30 these characteristics are adapted to vases' intended function (*inter alia* Bonaventure,
31 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
33 multiplied, during the late 1980s and early 1990s (Bronitsky and Hamer, 1986; Schiffer
34 *et al.*, 1994; Skibo, 1990; Young and Stone, 1990).

35 From the raw material to the surface treatments, the *chaîne opératoire* of ceramics
36 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
39 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
45 on how it was effectively used during its “life”, the study of which, has been conducted
46 by use-wear and residue analysis (Charters *et al.*, 1993; Condamin *et al.*, 1976; Drieu,
47 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

50 As part of our broader study on Prehistoric ceramics in the Mediterranean area (South
51 of France [3200-2300 B.C.] and Crete [3200 - 1100 B.C.]), we have investigated the
52 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
55 modifying the surface microtopography and suppressing micro- porosities, the potter's
56 technical actions also alter the vessels technical properties. Among various surface
57 treatments that have been identified, smoothing, burnishing and polishing can be
58 considered the most common (Balfet, 1966; Lepère, 2009; Lepère, 2014; Martineau,
59 2010; Rice, 1987; Rye, 1981; Shepard, 1956). Ethnoarchaeological investigations and
60 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-
63 Fedoroff, 2017; Rye, 1981; Shepard, 1956). Based on visual and tactile criteria, such
64 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
67 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
69 waterproofing coating cannot reach boiling temperature. However, it should be noted
70 that the goal is not always to reach a boil but can be to simmer the content. To achieve
71 this, coating is not necessary (Skibo, 1992). Their identification in the archaeological
72 record can be achieved under certain condition, using gas chromatography/mass
73 spectrometry analysis (Drieu, 2017). These types of studies are rarely applied in the
74 archaeological record although ethnographic investigations regularly highlight the
75 omnipresence of organic materials in pottery surface treatments (Gosselain, 2002;
76 Livingstone Smith, 2010; Schiffer, 1990). Moreover, distinguishing organic coatings on
77 archaeological vases from organic deposits related to the vases' function remains a
78 challenge.

79 The establishment of a reference database of surface treatments applied to flat clay
80 tablets, aims firstly at identifying signatures of organic and mineral surface treatments
81 at a microscopic scale; secondly, characterising surfaces' performance characteristics

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

84

85 **1. Methodology**

86 **1.1. Insights on various surface treatments in the prehistoric Mediterranean**

87 Pre-firing surface treatments such as smoothing, burnishing and slip coating, regularly
88 identified in the archaeological record, were applied with tools available during the
89 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).

92 The organic materials we used for our experiments are all attested by residue analysis.
93 Beeswax (Decavallas, 2011; Regert *et al.*, 1998), olive oil (among others Beck *et al.*,
94 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
98 treatments, of plant and animal origin, will be investigated in a broader study (see
99 discussion).

100 **1.2. Experimental material**

101 12 tablets, 5 square centimetres wide and 1.5 centimetres thick, were made for the
102 purposes of this experiment. We used raw materials found in karstic areas around the
103 Mediterranean: natural clay, sourced from Saugras (Hérault, France) and carbonate
104 temper (calcite and aragonite) from a cave in Saint-Pons-de-Thomières (Hérault
105 France).

106 After being cleared of its impurities, the clay was sorted. The temper was crushed and
107 sieved, to retain only grains thinner than 1 millimetre. Eventually, the temper was
108 added to the extent of 25% of the clay mass. The tablets were fired at 750°C for four
109 hours in an electric kiln, and thus under a controlled atmosphere. The temperature was
110 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
112 coherent for prehistoric firings.

113

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

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

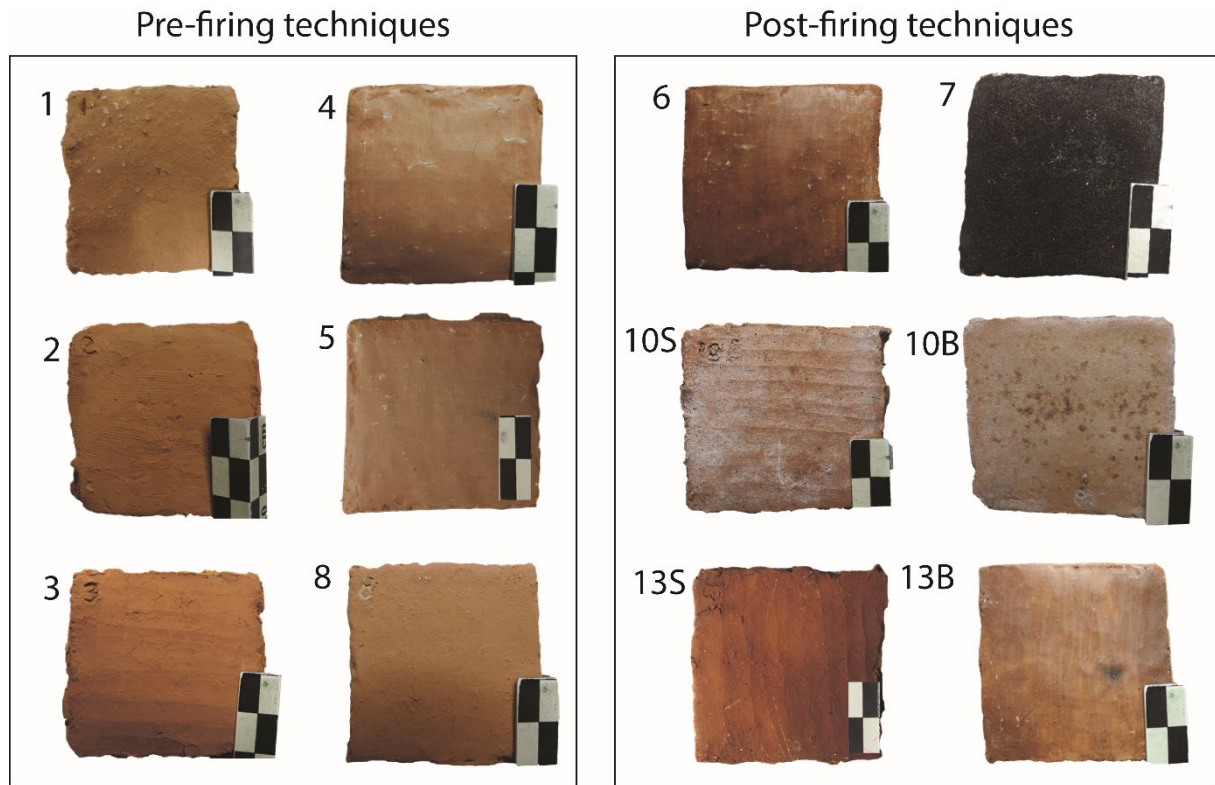


Figure 1: The experimental tablets.

N°	Tools	Prefiring treatment	Post-firing treatment	Technical gesture (post-firing)	Group
1	Wet hand	Smoothing	∅		I
2	Cloth	Smoothing	∅		I
3	Wood	Smoothing	∅		I
8	Slip coating	Smoothing	∅		II
4	Pebble	Burnishing	∅		III
5	Pebble, leather	Burnishing	∅		III
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	III
10B	Pebble	Burnishing	Milk on red hot surface	Dipping	IV
13S	Pebble	Smoothing	Beeswax	Smearing	II
13B	Pebble	Burnishing	Beeswax	Smearing	IV

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

135 **1.3. Macro-scale surface measurement with a confocal rugosimeter**

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

147 **1.4. Multi-scale analysis: the method of continuous wavelet transform**

148 This multi-scale analysis characterises the morphology of striations and the micro
149 roughness of polish. Observation of these marks on stone vases and tools has for
150 example been used to identify identify the abrasives employed as well as lubricants
151 and their viscosity (Procopiou *et al.* 2013; Boleti *et al.*, 2016).

152 As the Newton's prism decomposes white light into its spectral components, the
153 wavelet transform decomposes a topographic signal into its different scales:
154 roughness, undulation, shape. This multi-scale decomposition allows to quantify the
155 decomposed scales with the roughness coefficient S_{Ma} , which reflects the arithmetic
156 mean value of the signal's amplitude for a given wavelength. The results is presented
157 as a S_{Ma} spectrum, which constitutes a multi-scale signature of the topographic signal
158 of the surface (figure 2).

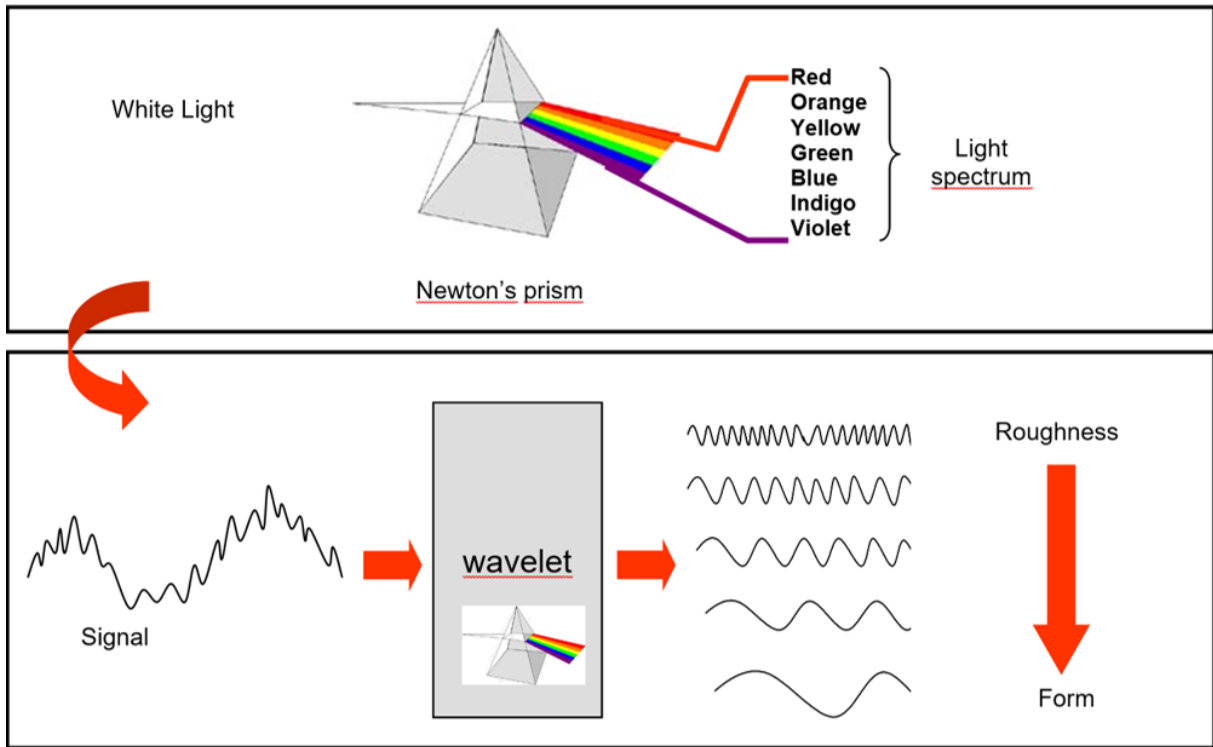
159 A wide range of wavelengths and amplitudes: $z(x, y) = f(L_i, L_j)$ are generally contained
160 in the distribution of the heights of a surface, corresponding to the three-dimensional
161 signal $z(x, y)$. Classifying wavelengths of roughness and waviness of a solid
162 conventionally depends on wavelength bands: shape (flat surface, cylinder, sphere: L
163 $> 2500 \mu\text{m}$), waviness ($500 < L < 2500 \mu\text{m}$), microscopic roughness ($1 < L < 500 \mu\text{m}$)
164 and nanometric roughness (a few $\text{nm} < L$).

165 The analysis of the geometrical aspects of the surface can be achieved using three
166 scales of observation: shape, waviness and roughness. The first and second of these,
167 permit the identification of manufacturing techniques. The third, allows the identification
168 of polishing techniques (Procopiou, *et al.*, 2011: 2528).

169 "The spectrum of multi-scale decomposition of a surface is obtained by the coefficient
170 S_{Ma} [...] representing the arithmetic mean value of all the scales of the surface
171 expressed as follows" (Lee 1997; Lee *et al.* 1998; Mezghani 2005; Zahouani & El
172 Mansori 2017).

173 In our case, we present the average wavelengths of three different measurements on
174 each surface.

175



176

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Figure 2: Multi-scale decomposition with the wavelet transform

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1.5. Needle-free drop deposition technique for contact angle

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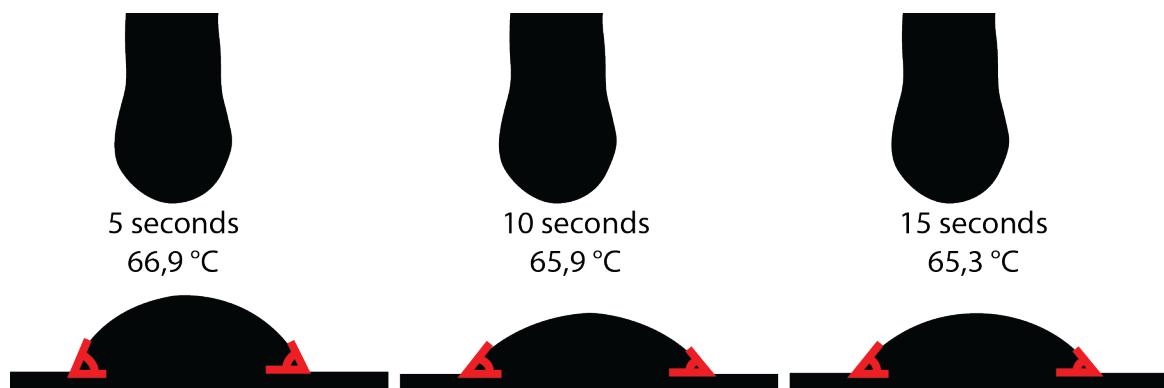
We also used drop shape analysis to evaluate the wettability of the surfaces, according to the surface treatment (figure 3). This apparatus uses a syringe that deposits a drop of a liquid substance on a plane surface (Stanimirova *et al.*, 2019). The duration of the deposition as well as the evolution 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.

185

186

187

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.



188

189

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

190 2. Results

191 **2.1. Surface measurement and characterisation**

192 Four groups of topographies can be observed (figure 4). The first two correspond to
193 the smoothed surfaces and the last two to the burnished surfaces. Surfaces in groups
194 I and III are plain, while surfaces in groups II and IV are coated. Our description of
195 states of surfaces will follow the terminology already defined by several scholars (for
196 example Buckley 1981).

197 It should be noted that no data could be recorded for tablets 7 and 10B, as their surface
198 was too dark or too white to be measured.

199 Surfaces in group I present an uneven topography. Striations are deep, unidirectional,
200 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
202 of clay.

203 Surfaces in group II also present an uneven topography, though slightly more regular.
204 Striations are less deep. They are unidirectional, linear, with a V-shaped cross section
205 and strongly connected. Edges have an irregular morphology. The coarse texture
206 results from the uneven spreading of clay. Coating reduces surface roughness by
207 decreasing asperities.

208 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.

211 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.

213 **2.2. Surface characterisation with the method of the continuous wavelet** 214 **transform**

215 The method of the continuous wavelet transform shows distinct signatures for
216 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
218 wavelengths than polishing while the addition of coating reduces the gap (figure 5).
219 These results therefore confirm the relevance of macroscopic characterization of
220 surfaces and provide, for the first time, roughness signatures of clay archaeological
221 surfaces depending on their type and degree of treatment.

222

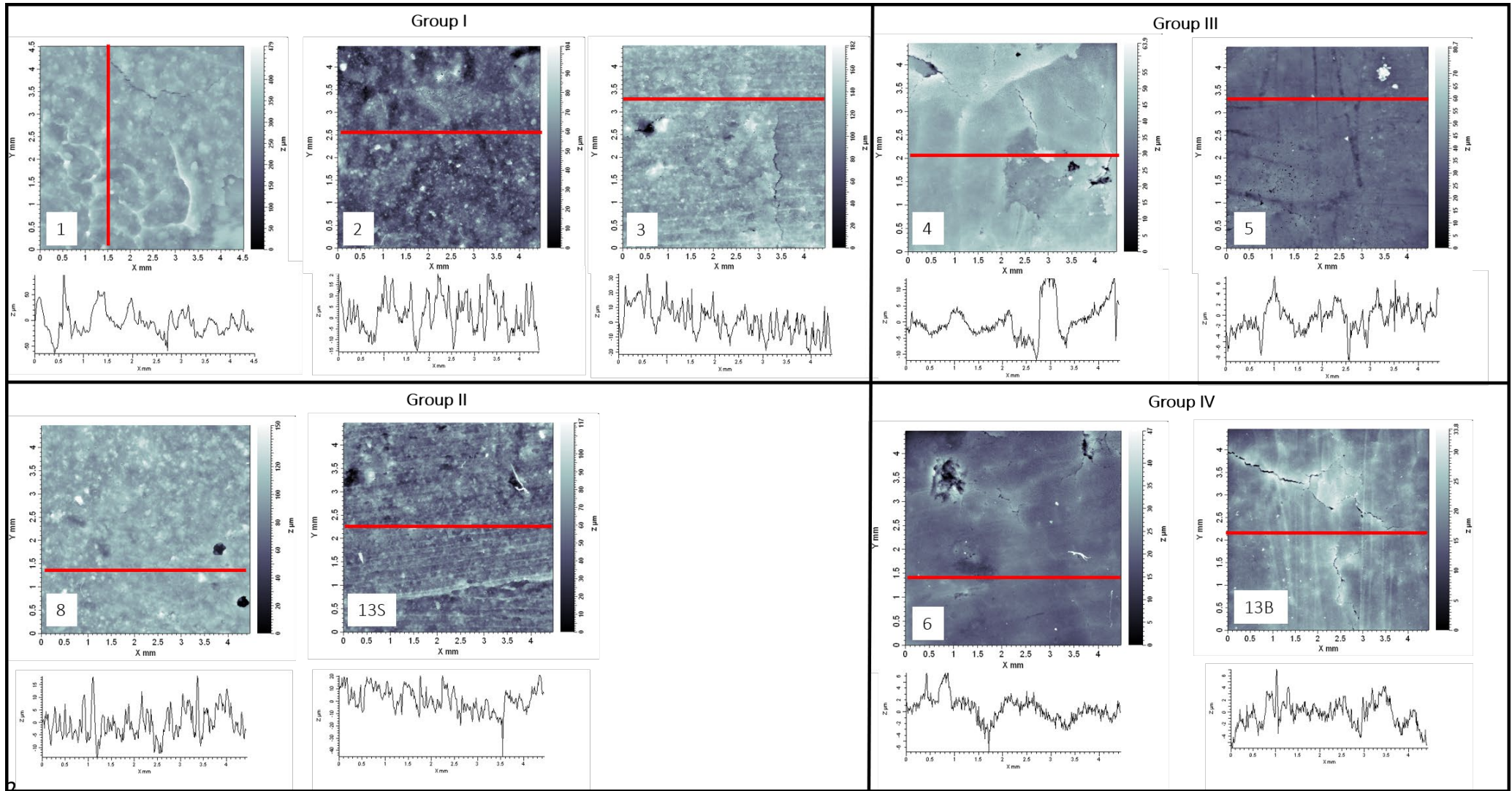
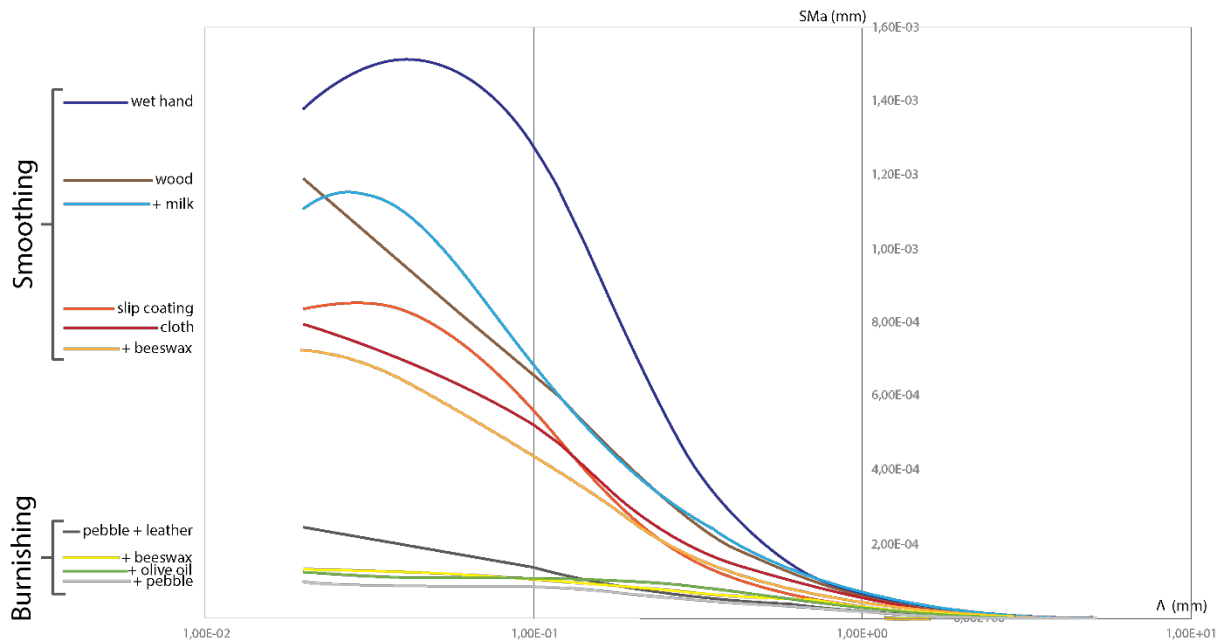


Figure 4: Four groups of topographies identified with the confocal rugosimeter. Profiles represent height and length in two dimensions.



225

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

228 **2.3. Surfaces' behaviours towards water drop deposition**

229 Water drop deposition showed that post-firing surface treatments have a clear sealing
 230 effect (figure 6), whatever the underlying surface, as had already been noticed by other
 231 scholars (Drieu, 2017; Gosselain, 2002; Lepère, 2014; Longacre et al., 2000; Schiffer,
 232 1990). It should be noted that no data could be recorded for tablet 7, as its porosity
 233 directly absorbed the drops.

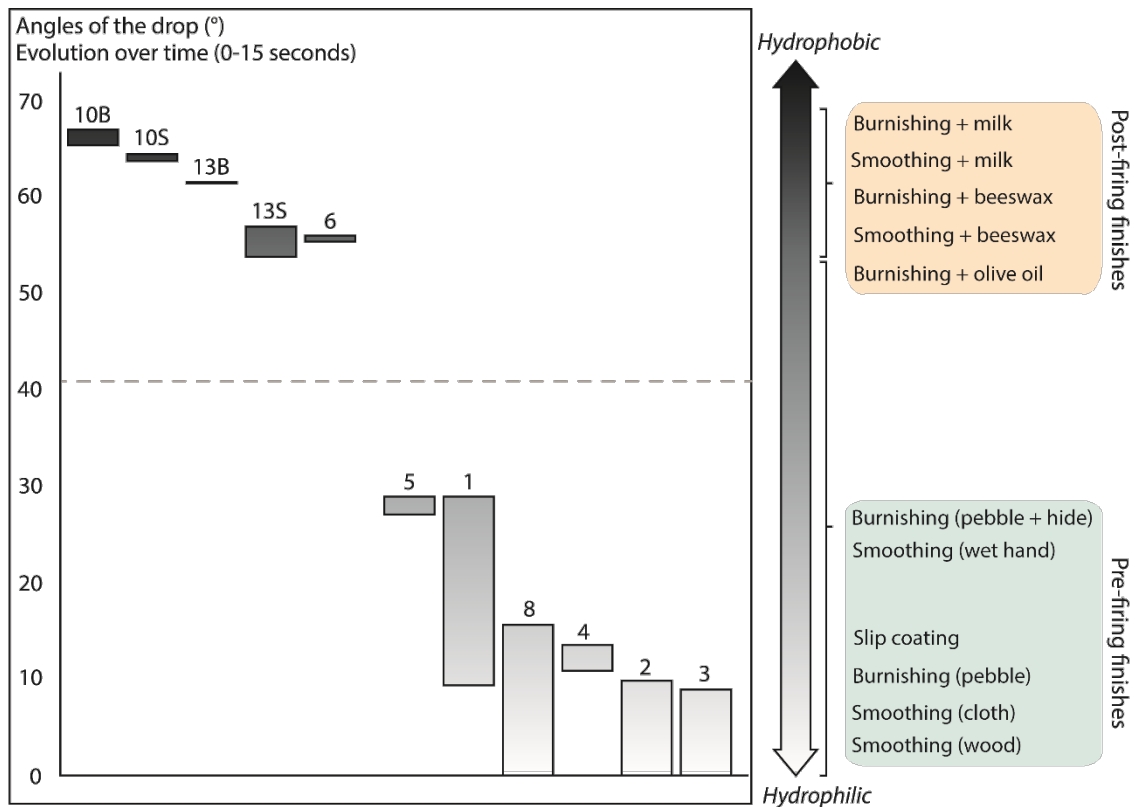
234 Angles of water drops were highest when deposited on surfaces coated with beeswax,
 235 milk and olive oil: ranging from 50 to 70°, they stood still over the span of 15 seconds
 236 and then held for at least 100 seconds.

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

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

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

247



248

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

251

252 **2.4. Surfaces' behaviours towards oil drop deposition**

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

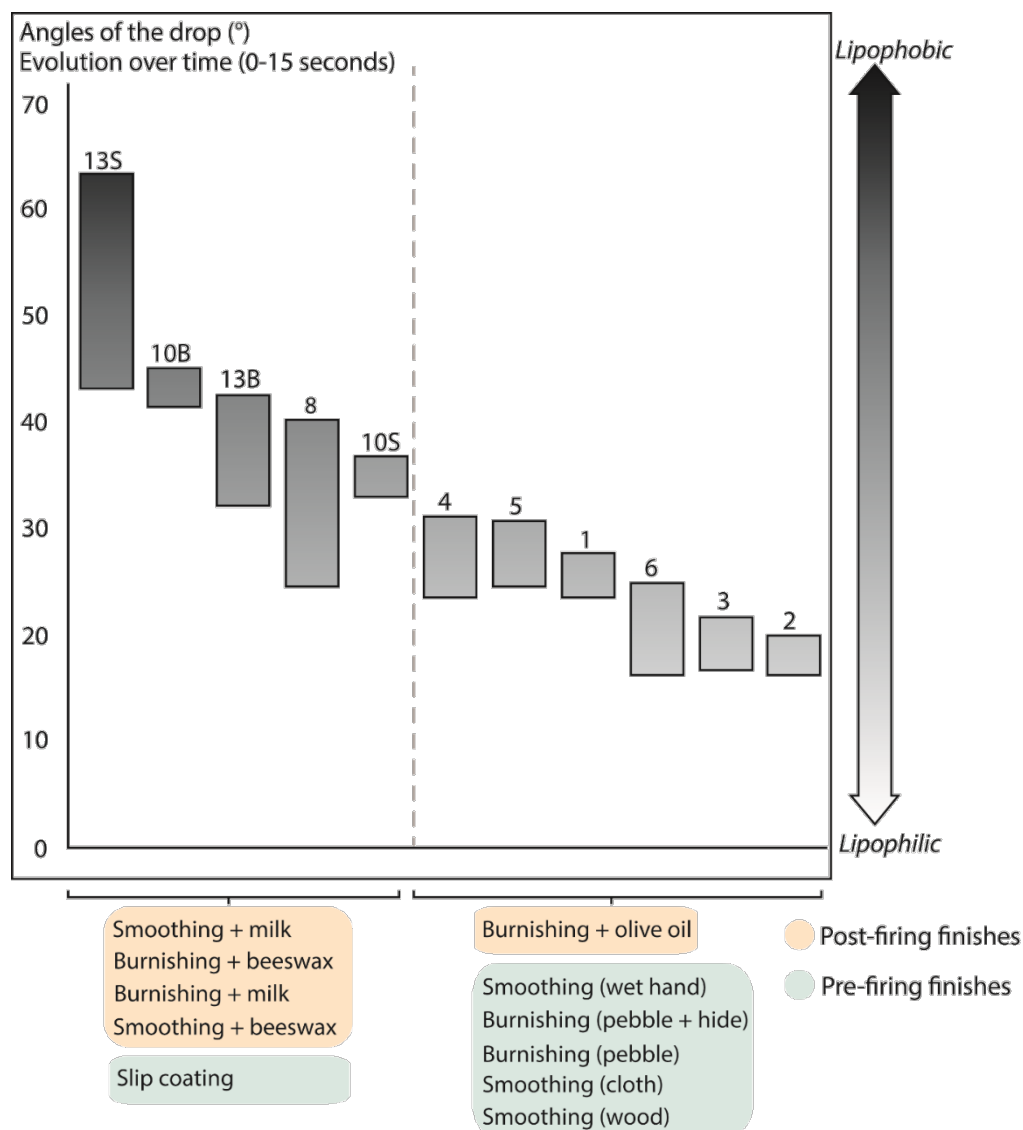
259 With the exception of tablet 6, post-firing surface treatments, along with slip coating,
 260 proved once again to have the best sealing properties, whatever the underlying surface
 261 (figure 7). Ranging from 63.2 to 36.5°, the angles ranged from 43.2 to 24.9° after 15
 262 seconds.

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

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

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

270 its viscosity as well as upon the state of surface on which it is dropped. In the case of
 271 post-firing coatings, partly composed of lipids, the oil drop mixes up with the coating.
 272 As such, the drop is not absorbed by the surface but spreads horizontally. It can be
 273 assumed that, after many uses, fatty contents would act as self-coating.



274
 275 *Figure 7: Results of the oil drop deposition on the experimental tablets. Rectangle length*
 276 *represents evolution over 15 seconds*

277
 278 **3. Discussion: Surface treatments and performance characteristics**
 279 **of clay vases**

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

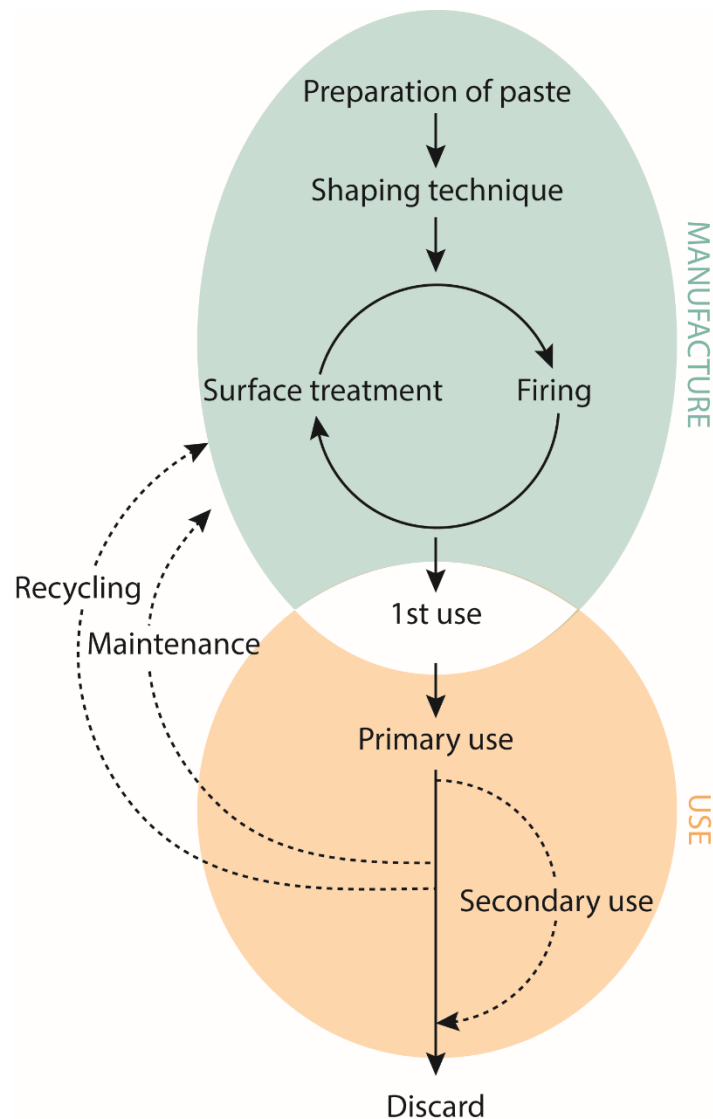
285 reference database still needs enlarging, especially with regard to organic materials.
286 We have so far focused on milk, wool, olive oil and beeswax but other materials such
287 as animal fats, birch bark tar, etc. should be considered in the future.

288 Roughness was measured with a confocal microscope and characterised by the
289 method of the continuous wavelet transform, providing, for the first time, a microscopic
290 signature of surface treatments. These reference data must be compared, in the future,
291 with those from the archaeological record.

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

301 Neither chemistry nor microscopic analysis can yet differentiate the initial motivation
302 behind the formation of organic residues, that is to say determine whether they reflect
303 an intentional coating or the content of the vase. However, prehistoric users may not
304 have differentiated between the two stages. Indeed, the first uses of a cooking pot can
305 aim at testing the integrity of the vessel, at removing or lessening the earthy taste and
306 waterproofing the container. Moreover, some studies suggest that boiling organic
307 material in a vase before use will not only waterproof it but will also significantly reduce
308 the time needed to reach boiling temperatures in the future (R. P. Evershed *et al.*,
309 1997; Skibo, 1992). By taking this into perspective, the first use and the last stage of
310 the *chaîne opératoire*, the organic coating, can in some case be superimposed (figure
311 8). A number of coatings can be obtained by applying organic material on a warm pot
312 or simply by warming it up inside the vessel (Gosselain, 2002; Harry *et al.*, 2009;
313 Henrickson and McDonald, 1983; Rye, 1981). In the case of edibles, the content can
314 potentially be consumed afterwards.

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



323

324

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

325

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

331

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

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

352

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

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554 Acknowledgements,

555 The authors wish to thank Alain Giosa and Christophe Petit (Université Paris 1 Panthéon-
556 Sorbonne, UMR 7041) for their kind provision of the kiln that fired the tiles. Their gratitude
557 also goes to the *Société Languedocienne de Préhistoire* for the provision of the clay and to
558 Euan Wall and Marilena Pateraki for having proof-read the text.

559