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Multi-purpose pots: Reconstructing early farmer behaviour at Lydenburg Heads site, South Africa, using organic residue analysis

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ABSTRACT

About 2000 years ago, Bantu-speaking people introduced an agro-pastoral lifeway into southern Africa and lived in northern and eastern lowland parts of the region by 700 CE. Also defined as the 'southern African Iron Age', pottery is the most common and diagnostic artefact category. The soil chemistry of several farming sites resulted in poor faunal preservation and especially within Mpumalanga Province, South Africa, the possibilities for dietary reconstructions based on bone assemblages is very limited. Furthermore, the majority of ceramic research on early farming communities (ca. 200–900 CE) has focused on typology and ethnoarchaeological interpretations. This study represents the first application of Organic Residue Analysis (ORA) applied to southern African early farming pottery to gain a deeper understanding of past human behaviour and subsistence patterns. We investigate the potential and evaluate future prospects of applying this method to southern African early farming pottery. A combined lipid biomarker (GC-MS) and compound specific isotope approach (GC-C-IRMS) was applied to pottery sherds ($n = 40$) from the Lydenburg Heads site (LHS), dating to the 7th century CE (Mzonjani facies) and 9th to 11th centuries CE (Doornkop facies), to test for lipid preservation, trace possible vessel use and dietary changes through time and embed the research data within the faunal and botanical record available. Our study provides first evidence for the processing of dairy products by early farmers in southern Africa. Both facies imply multi-purpose functionality of the ceramics tested, demonstrated by the mixing of animal fats and plant oils. The obtained ORA data from LHS confirms the processing of ruminant carcass fats within both occupation facies, likely representing domestic animal fats, as well as the processing of wild non-ruminant carcass fats within the earlier Mzonjani facies. The identification of ricinoleic acid in two vessels of the Doornkop facies, provides evidence for potential medicinal use. Preliminary data also indicate that dung could have been involved in different forms of pottery sealing and/or mending during both occupation facies. In this study, we begin to explore biomolecular evidence for dietary choices made by early farmers in southern Africa, shedding light on previously unexplored key products.

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1. Introduction

Around 2000 BP, the human population of southern Africa comprised groups of hunter-gatherers and, in the western and southernmost parts, pastoralists. From the third century CE, Bantu-speaking, metal-producing farmers expanded into the summer rainfall zone of northern and eastern southern Africa and promoted a rapid spread of new practices, such as livestock keeping, crop cultivation and permanent settlements. For settlement, they favoured riverside locations in wooded (savanna/bushveld) country that provided year-round sweetveld pasture and fuel for industrial and domestic use (Maggs, 1980, 1994-95). The earliest sites tend to occur in the vicinity of iron-ore sources, suggesting that ore prospecting was an important component of the expansion of farmers southward of eastern Africa (e.g., Whitelaw and Moon, 1996). Iron tools, most especially hoes, were essential to their agricultural subsistence. For this reason, archaeologists have often described these farmers as 'Iron Age' in contrast to 'Stone Age' hunter-gatherers, who continued to live as neighbours in varying forms of interaction with farming communities (Mazel, 1989; Wadley, 1996). Generally, archaeologists have divided the Iron Age into Early (first millennium CE) and Late (second millennium CE) periods, except for a Middle Iron Age (900–1300 CE) for sites and processes related to the origin of the Zimbabwe Culture in and around the Shashe-Limpopo confluence area (Huffman, 2007).

Subsistence-related data for first millennium farmers come from sites from Zambia southwards. The crops identified are the grains bulrush and finger millet and sorghum, cucurbit species such as the bottle-gourd, and the pulses cow-peas and ground-beans (Fowler et al., 2000; Huffman, 1979; Klapwijk, 1974; Maggs, 1984; Maggs and Ward, 1984; Olatoyan et al., 2022, p. 54; Robertson, 2000). Like the rest of sub-Saharan Africa, the food preparation techniques of these first-millennium farmers would have been informed by what Haaland (2007, p. 169) labelled the cooking pot syndrome. In this, pot cooking is favoured over oven baking for practical and symbolic reasons, and most food is consumed in the form of porridge, stew and beer. These preferences continued into the recent past, during which grains were processed into a variety of forms ranging from gruels to porridges to bread-like or dumpling forms to, most popularly, a stiff or crumbly but kneadable material. People generally ate these grain-based foods with relishes or gravies made from domesticated or wild leaves, cucurbits, pulses and meats, or with wild fruit juices or sour clotted (fermented) milk (e.g., Krige, 1962, p. appendix IV; Quin, 1959). Grain was also the principal ingredient for beer, which was probably brewed mainly from millets, with sorghum preferred for harder foods (Bryant, 1967, p. 274 ff.).

Faunal samples are usually dominated by domestic livestock, with sheep numbers greater than cattle. Goat remains are considerably fewer than sheep. Some interpretations argue that cattle became significant only late in the first millennium (Badenhorst, 2011, 2018; Maggs and Whitelaw, 1991; Plug, 1989; Voigt, 1986), but larger faunal assemblages indicate consumption of more beef than mutton (e.g. KwaGandaganda and Mamba in KwaZulu-Natal as well as Bosutswe and Nqoma in Botswana), either boiled or roasted (Beukes, 2000, pp. 68, 122; Maggs, 1994, p. 176). Moreover, numerous sites preserve remnants of livestock pens around 25 m in diameter; these most likely penned cattle (Huffman, 1993). Ethnographic accounts suggest that their subsistence value, apart from occasional meat feasts, came regularly from their milk, which people commonly fermented for consumption in bottle-gourd containers. People in some farmer groups also drank goats' milk (Quin, 1959, p. 226). Milk, soured or fresh, would have provided nutritional cover during the 'lean months' at the transition from one agricultural year to the next, when supplies ran short and fresh produce was unavailable. Faunal analysts have, in recent years, identified the occasional chicken bone in first millennium farmer settlement contexts, but archaeological and ethnographic evidence gives these birds limited subsistence value (Beukes, 2000, pp. 92–93; Plug, 1996, p. 517).

There is considerable evidence for the use of wild resources. Numerous shell middens on the beaches of the east coast of South Africa are the product of first millennium farmers harvesting mainly brown mussels. Evidently, parties from settlements up to 30 km inland participated in the harvesting, most probably during spring low tides (Horwitz et al., 1991; Punyadeera et al., 1997, 1999). Sites inland of Natal Bay (Durban) also contain the bones of marine fishes, which were possibly acquired from farming communities operating fish traps in the bay. By contrast, freshwater molluscs and fishes do not seem to have had much subsistence value, especially considering the proximity of settlement sites to rivers (Whitelaw, 2009). Apart from molluscs, which included Achatinid land snails used for bead making and possibly food, invertebrates are not represented in the archaeological record. Yet ethnographic accounts record extensive subsistence use of caterpillars, locusts and grasshoppers, termites, and other invertebrates. Invertebrates were likely gathered for relishes and as such would have supplied people with high quality protein and fat (Quin, 1959). Indeed, the delicious flavour of termites and their abundant emergence at the onset of spring rains no doubt contributed to their ritualization among some groups of African farmers as 'the food of chiefs' (e.g., Hammond-Tooke, 1981, p. 9).

Animals hunted by first millennium farmers range widely from elephants and hippopotamuses, through bovid species of various sizes, primates and carnivores, a few bird species, monitor lizards and crocodiles, to tortoises (e.g., Badenhorst, 2015; Beukes, 2000; Brown, 1981; Grody, 2016a, 2016b; Plug, 1996, 1989, 1988; Plug and Voigt, 1985; Voigt and Plug, 1984, 1981). The most common species include wild grazers, such as buffalo, zebra, and wildebeest, as well as mixed feeders, such as impala. Their subsistence significance is generally limited. Exceptions occur in the Lowveld of east and northeast Mpumalanga Province, South Africa, where the meat weight represented by hunted animals in the faunal samples is far greater than from livestock. Also, the catastrophic mortality profiles of several wild herd animals indicate communal hunting (Plug, 1989). In the case of at least two sites, these and other butchering data most likely reflect the specialised production of dried and perhaps salted meat for transport elsewhere (Grody, 2016a, 2016b).

Farmers also exploited wild plant foods and possibly fungi (e.g., Richards, 1951, pp. 36, 41), though the archaeological evidence is limited and often unreported (but see Steyn and Antonites, 2019) for the Middle Iron Age). Sites in the Thukela and Mngeni valleys yielded charred and uncharred marula stones, typically with the opercula 'popped' for extraction of the kernels (e.g., Maggs, 1984). Ethnographic accounts suggest that kernel extraction took place in the course of using the fruits to create an intoxicating beverage (e.g., Cunningham, 1988, p. 435; Quin, 1959, p. 247). These accounts indicate farmer use of a very wide range of fruits, 'spinaches', roots and tubers, and tree saps. Although wild fruits were often eaten at source, wild food types were also gathered and processed in the homestead. In some cases, the processed foods were enhanced with ingredients such as honey (e.g., the dried and ground pulp of *Strychnos* fruits). In summary, wild foods would have given first millennium farmers a critical nutritional boost, adding variety and supplementing their diet with vitamins, oils, amino acids and minerals lacking in foods solely from domestic crops and animals. Additionally, they contributed to food security in the annual lean months and during periods of drought (Cunningham, 1988). Yet, the archaeological record so far contains only the 'bare bones' of the Iron Age subsistence base. Many resources and preparation strategies may never be represented by traditional archaeological food data. Here, Organic Residue Analysis (ORA) offers an alternative investigative technique, characterising vessel content to attempt to reconstruct the availability and use of plant and animal resources, while complementing the rich ethnographic accounts. ORA is a well-established method and has been routinely used for over four decades to characterise lipid residues trapped within the porous fabric of ceramic vessels, providing valuable insights into the consumption and preparation of a wide range of plant and animal resources, vessels function (e.g. cooking, storage), the movement of goods, repair and sealing of vessels (Evershed, 2008a; Evershed et al., 1992). To date, lipid analyses have rarely been applied to sub-Saharan African material, though commonly undertaken elsewhere. While ORA studies (Copley et al., 2004; Dunne et al., 2019, 2021; Fewlass et al., 2020; Grillo et al., 2020; Hopper et al., 2023; Quintana Morales et al., 2022) have proved the feasibility of applying this methodology to Neolithic, Pastoralist, hunter-gatherer and West African early farmer contexts, ORA has not yet been applied to an early farming context from southern or eastern Africa. Specifically regarding South Africa, the faunal preservation is patchy depending on location and hence burial conditions, and very few sites have provided significant botanical assemblages (Becher, 2021). Here, ORA has the potential to fill some of these gaps in our understanding about past human subsistence.

Although pottery is the most common and diagnostic artefact category, up until today little is known about the relationship between vessel features, such as shape, decoration or manufacture, and vessel function as the majority of ceramic research on early farming communities has focused on typology and ethnoarchaeological interpretations. Ethnographic studies (Fowler, 2006; Huffman, 1972; Quin, 1959) can be valuable tools to understand past practices, however, similarities between isolated observations on material culture with selected ethnographic examples lack in coherence with other social, religious, and economic aspects (Hodder, 2012). Hence, an understanding of pottery function across space and time in southern Africa could be a valuable tool for reconstructing past behaviour and the analysis of organic compounds, such as lipids, can provide strong evidence for the interpretation of the life histories of vessels by identifying e.g. specific animal, plant and/or cooking markers.

Here we report on the ORA results of potsherds from the first millennium CE Lydenburg Heads site (LHS). LHS is an open-air site situated in the Sterkspruit valley on the southeast edge of the town of Lydenburg in the province of Mpumalanga, South Africa (Fig. 1). K.L. von Bezing found artefacts scattered across LHS in the 1950s which were collected in the 1960s. His collection included fragments of the now iconic seven terracotta heads (Inskeep and Maggs, 1975). The site was then excavated in the 1970s by Mike Evers (1982). LHS is impacted by fairly severe gully and sheet erosion that had uncovered both the head fragments and exposed many deep pit features. Evers's (1982, p. 17) most complete stratigraphic sequence comprised a shallow horizon of secondarily deposited sherds and bone over truncated pits. He excavated 12 pits (Fig. 2), which contained assorted stones, sherds, daga, bone and charcoal, sometimes in a 'khaki ash' matrix; subsequent research elsewhere in South Africa suggests that the khaki ash might have been dung (e.g. Huffman, 1993). Similar pits occur on first millennium farmer sites from Tanzania to the Eastern Cape in South Africa. They were most probably originally storage pits for crop foods that were subsequently filled with waste materials. Some, even many, pit fillings probably derived from ritual events (e.g., Maggs and Michael, 1976; Whitelaw, 1993).

The LHS ceramics came from the surface, the uppermost horizon and from pits. The pits provided the only secure context. The ceramics consist of pots with either everted or slightly taller, upright rims, and both open and inturned (restricted) bowls (Evers, 1982, Figs, 8–15). As is typical of this period, almost all pots are decorated. At LHS, decoration occurs as bands of incised hatching, cross-hatching and herringbone, rows of impressions, and various isolated motifs applied to the rim, neck area, shoulder and body of vessels. Simple open bowls are typically undecorated, but decoration was applied to the inturned bowls (Evers, 1982, Fig, 15). This different treatment of bowl types suggests different functions, because ethnographic research indicates that the degree of decoration on an item of material culture can relate to the context of its use (Armstrong et al., 2008; Braithwaite, 1982; David et al., 1988). Thus, at LHS, the open, undecorated bowls were possibly used for cooking and the decorated, inturned bowls for serving food (perhaps meat and vegetable relishes). The LHS pots are all essentially similar in shape. The variation that exists in rim shape relates to different and chronologically distinct ceramic traditions rather than function (Whitelaw, 1996). Function, therefore, was most probably related to the

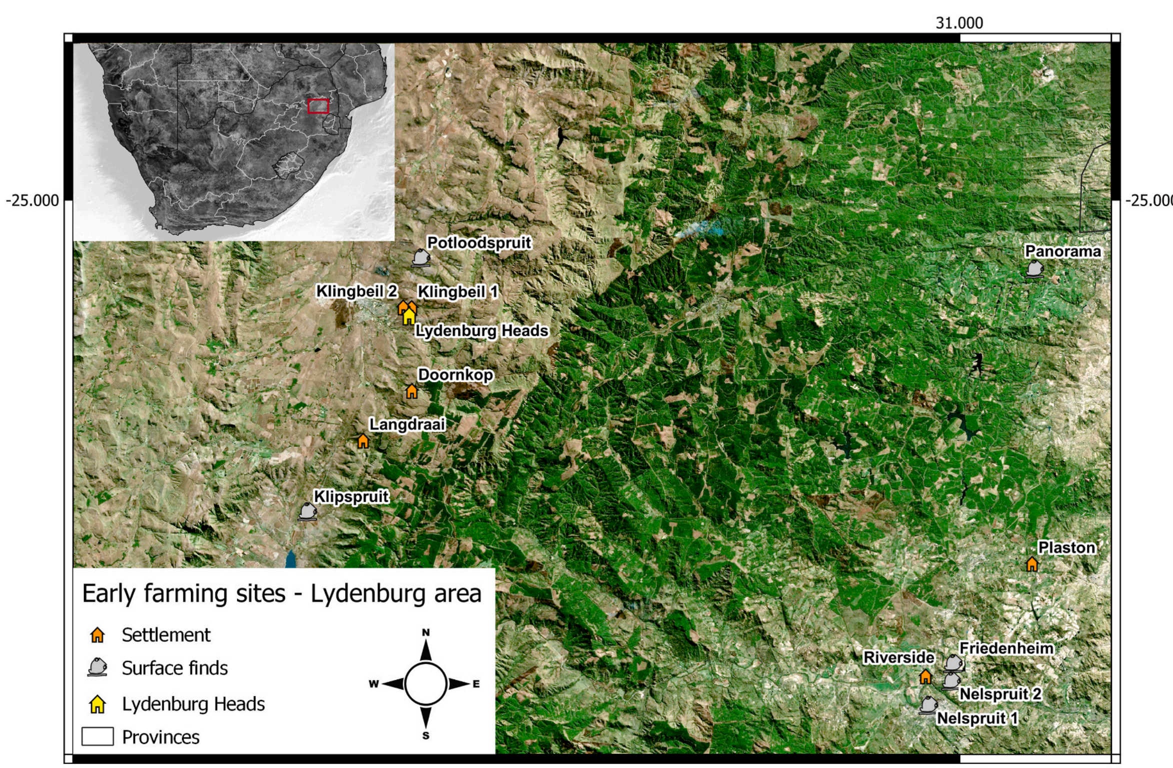

Fig. 1. Location of Lydenburg Heads site (in yellow) in South Africa and other early farming sites within close vicinity. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

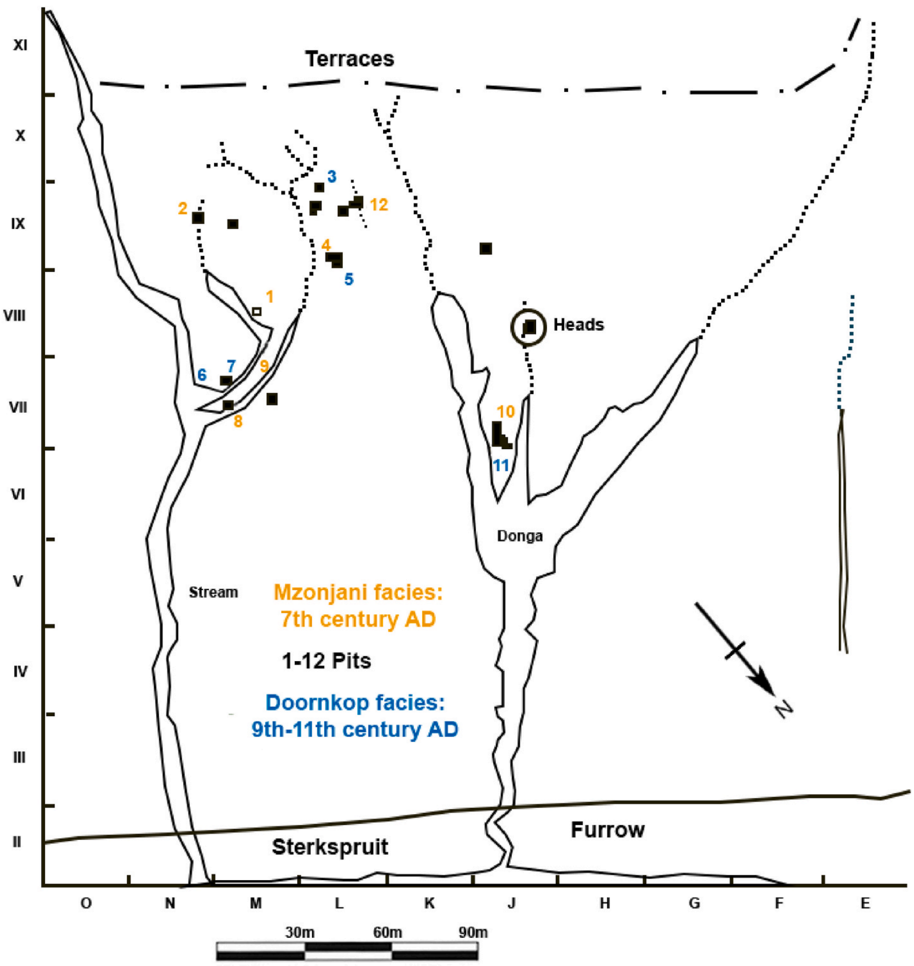

Fig. 2. Site plan showing excavations and locations of features. Allocation of Whitelaw's Group 1 (Mzonjani facies) and 2 (Doornkop facies), modified after Evers (1982) and Whitelaw (1996).

size of pots. Pots were most probably used for cooking, brewing beer and other beverages, transport of foodstuffs and water, serving and storage. Storage and brewing vessels were most likely larger than others, vessels that were carried for serving or transport of foodstuffs must have been at the smaller end of the size spectrum, while a variety of pot sizes might have been used for cooking. Some pots, and possibly inturned bowls, must have been used for medicinal preparations. Additionally, pots were used in off-site contexts, such as in salt production (Antonites, 2013, 2016; Evers, 1979) and shellfish harvesting (Horwitz et al., 1991).

Evers (1982) placed all the ceramics from LHS into his Lydenburg facies. The material was reanalysed by Gavin Whitelaw (1996), who recognized that it contained two ceramic facies (Fig. 2). The first dates to the 7th century CE and is an Urewe Tradition style called Mzonjani. The later one is a Kalundu Tradition style called Doornkop and probably dates from the 9th to 11th centuries CE. So far, only the Mzonjani facies has provided absolute dates with ^{14}C-dates to cal. 640–680 CE (Pta-1634, charcoal) stratified from pit 1 and cal. 605–660 CE (Pta-328, charcoal) from the surface close to pit 8 (Evers, 1982).

No macrobotanical food remains were reported from LHS, or any other first millennium site in the area. Furthermore, the acidic nature of the soil at the site and surrounding area (pH = 5.3) resulted in poor bone preservation (S1, Tables S1–S2 and additional information). The original faunal report from LHS does not differentiate between the two occupation facies (Evers, 1982). Following Whitelaw's (1996) facies' identification, it was possible to re-evaluate the faunal evidence and separate most of the data into the two ceramic traditions (Becher, 2021) (see S1, Table S1-S2). *Ovis/Capra* species were securely identified only in the Mzonjani facies, whereas domestic cattle are present in both facies but more abundant in the Doornkop facies (Becher, 2021). As mentioned above, this pattern of caprines outnumbering cattle has already been identified at various early farming sites (Badenhorst, 2008).

Here, we present the results on lipid preservation in pottery from LHS, discuss possible dietary changes and differences in vessel use through time by comparing the facies present at the site while embedding the research data within the available zooarchaeological, archaeobotanical, and ethnographic record. In order to address these research questions, a review of the published faunal and botanical record was conducted, which were used to interpret possible sources of the lipid data. The geographical framework of this comparative literature review included data from LHS as well as other early farming sites in Mpumalanga, Limpopo, North-West, and Eswatini (S1, Fig. S1, Table S3).

2. Materials and methods

In the current study, we investigate 40 organic residue samples from LHS (n = 20 of each facies) using a combined lipid biomarker and compound specific isotope approach to be able to test the lipid preservation and to compare the occupation facies (S1, Figs. S2–S9). Of the Mzonjani facies, one vessel was sampled at the shoulder and base area (samples LHS-26-27), as were two vessels of the Doornkop facies (samples LHS-21-22 and LHS-36-37). In addition, four soil control samples were collected from across the site to test for exogenous contamination (S1, Figs. S10–S11), and the exterior surfaces of the vessels were also tested. The pottery samples were selected based on reconstructed shapes and their possible use following ethno-archaeological interpretation. Although from a different region and temporal context, the study conducted by Fowler (2006) served as a helpful tool to choose ceramics from specific pottery classes, while focusing on pots that cluster under the categories of 'cooking & brewing', 'storage' and 'medicinal & ritual' (Fig. 3). Unfortunately, no clearly identifiable sherds falling under the latter category could be found at LHS, hence our analysis focused on cooking pots and storage vessels. The vessels were sampled at the iThemba LABS (Laboratory for Accelerator Based Sciences) in Johannesburg, South Africa. From each sherd, 2 g of ceramic powder (Interior Layer 2, In L2) was collected with a Dremel 4000 drill fitted with tungsten carbide drill bits. Prior to sampling, the surface of each sherd was cleaned with the drill to remove any surface contamination.

Solvent extraction was carried out following published protocols (Rageot et al., 2019) at the Organic Residue Laboratory at the University of Tübingen, Germany. To avoid any contamination arising during laboratory work, aluminium foil and glassware were sterilised at 500 °C for 6–8 h in a muffle furnace. All metal objects, such as tweezers and drill bits, were solvent cleaned for 15 min in a Dichloromethane:Methanol solution (DCM:MeOH, 2:1, *v*:*v*). The extractions were carried out with HPLC grade solvents and a method blank was processed with each batch which was extracted to monitor for laboratory contamination. The samples were analysed using gas chromatography-mass spectrometry (GC-MS) and, if applicable, GC-Combustion-Isotope Ratio-MS (GC-C-IRMS). Detailed descriptions of the methods can be found in the supplementary information (S1).

3. Results

Residues were considered significant when >5 μg^{-1} g of lipid were obtained (Evershed, 2008b). Within each pottery tradition, 13 out of 20 samples yielded a significant amount of residue with a mean of 278.2 μg^{-1} g for the Mzonjani samples and 282.3 μg^{-1} g for the Doornkop samples (Fig. 4; S1, Tables S4–S5; S2, Tables S1–S2).

No laboratory introduced contamination could be identified. The four soil control samples (LHS-1a, LHS-1b, LHS-1c, and LHS-2) yielded similar profiles of detected compounds (S1, Figs. S12–S15). The most abundant compounds were sterols and stanols deriving from plant origin, namely campesterol, β-sitosterol, stigmasterol and stigmastanol. Cholesterol was present in only trace amounts. Furthermore, long-chain even-numbered alcohols (C_{26}–C_{32}) were abundant, as well as long-chain *n*-alkanes. These are consistent with plant and animal detritus (Eglinton and Logan, 1991; Heron et al., 1991). The soil control samples contained fatty acids in trace amounts. Sample LHS-1b, which was taken from an intact pit exposed in a donga (South African term for an erosion gully or furrow) wall, represented an exception; this included high amounts of $C_{16:0}$ followed by $C_{18:0}$ and $C_{18:1}$. This pattern in combination with the long-chain even-numbered alcohols as well as sterols and stanols supports a plant lipid origin (see e.g. Dunne et al., 2017). No wax esters were detected in any of the soils but the long-chain alcohols and fatty acids represent their degradation products. Small amounts of sugars were detected. Mycose was abundant, indicating microbial biomass activity through fungal input (Jaffé et al., 2006; Simoneit et al., 2004).

All these compounds are typical of soil samples and demonstrate the input of organic matter from plants and animals and microbial activity by fungi and bacteria in the soil (Heron et al., 1991). Based on the soil

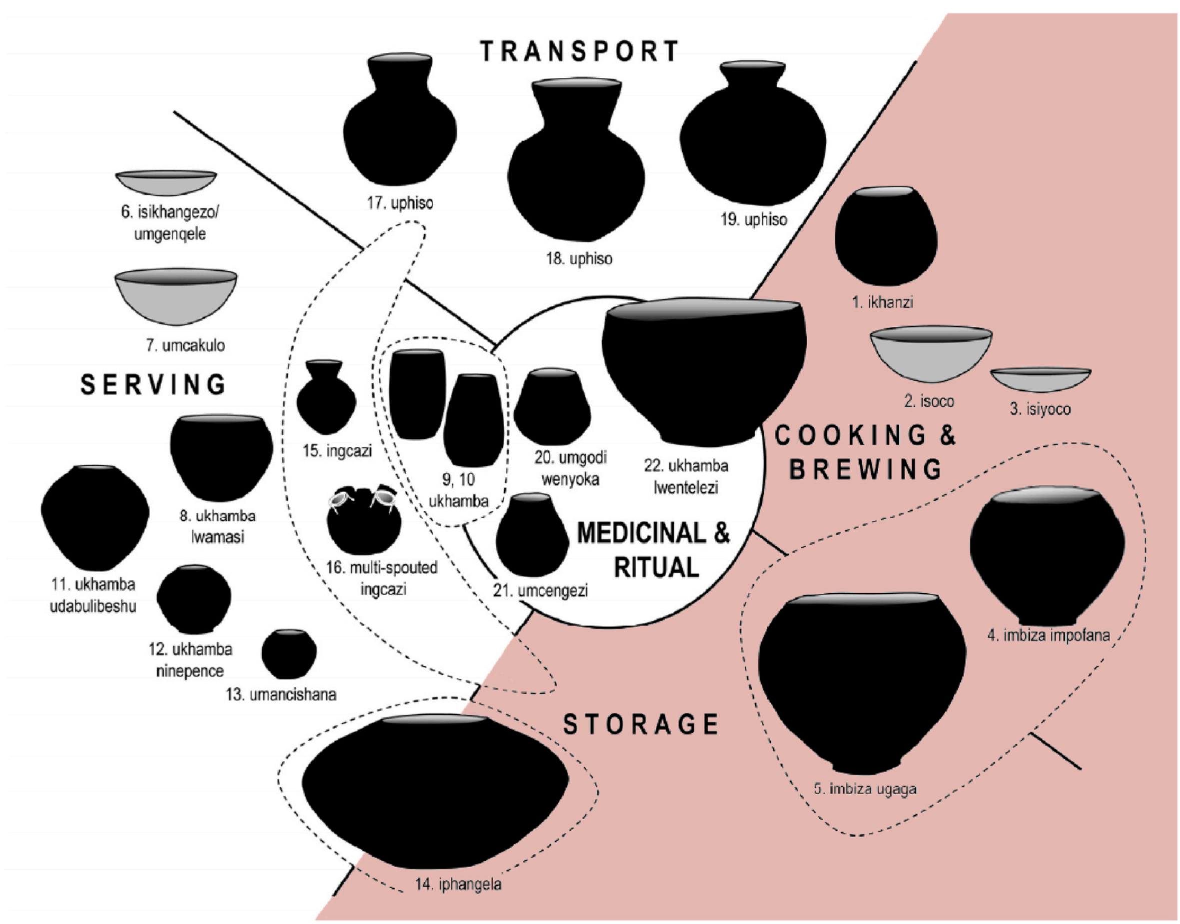

Fig. 3. Distinctive functional classification of Zulu ceramic containers (after Fowler 2006, Fig. 2). In red: focus on pottery clusters for current study. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

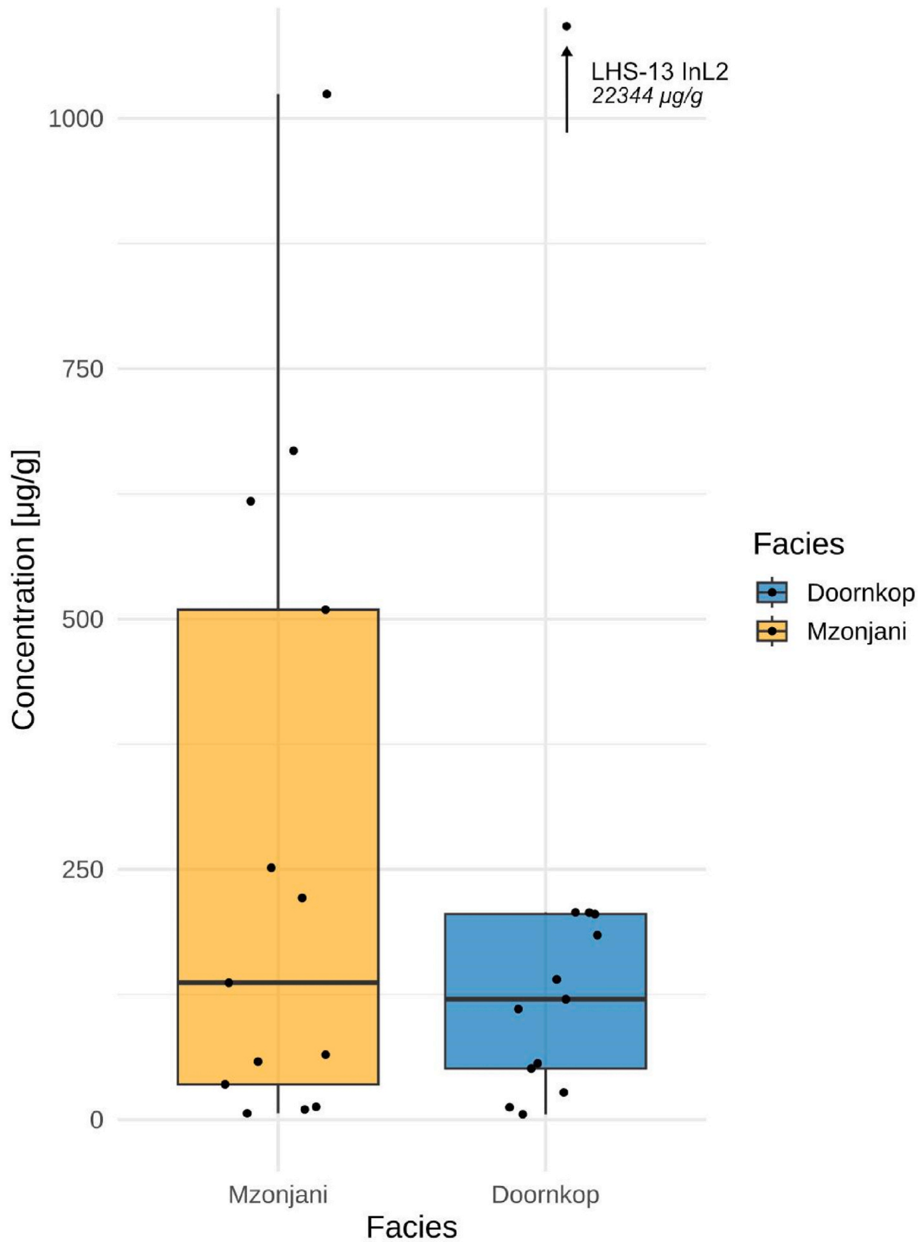

Fig. 4. Box plots of interpretable samples from LHS visualising lipid concentrations showing first [Q1] and third [Q3] quartile, median and outliers.

control samples in combination with sherds showing a negligible quantity of lipid, leaching from the burial environment seems highly unlikely. This indicates that the extracted lipid molecules represent the actual archaeological vessel use.

3.1. *Mzonjani facies (Urewe Tradition)*

The Mzonjani samples provided a wide range of compounds pointing towards an array of exploited resources and mixing of these (see S1, Figs. S16–S17; S2, Table S1).

The Mzonjani samples contained saturated fatty acids, ranging from $C_{8:0}$-$C_{28:0}$, with branched *iso-* and *anteiso*-fatty acids of $C_{13:0}$, $C_{15:0}$ and $C_{17:0}$. Only $C_{16:1}$ and $C_{18:1}$ were identified as unsaturated monocarboxylic acids and occurred in small quantities. The presence of $C_{18:1}$, could possibly originate from plants (Regert, 2011), however, unsaturated octadecanoic acids can also derive from an animal origin, including carcass and dairy fats (Regert, 2011). Due to biohydrogenation of dietary fats in the rumen, multiple positional $C_{18:1}$ isomers paired with $C_{15:0}$, $C_{17:0}$, and branched *iso-* and *anteiso*-fatty acids, which are linked to bacterial synthesis of ruminant animal guts, may indicate ruminant fat (Regert, 2011).

Ten out of 13 samples yielded acylglycerols. The identified triacylglycerols (TAGs) ranged between C_{46} and C_{54} with even-numbered TAGs C_{48}, C_{50} and C_{52} being the most common. The correlated degradation products could be observed within the DAGs' and MAGs' profile of the respective samples. The TAG distribution can hint at the vessel content. Ranges between C_{42} and C_{54} originate from ruminant bovine adipose fat, whereas the TAG ranges for ruminant ovine adipose and non-ruminant porcine fat fall between C_{44} and C_{54} (Mirabaud et al., 2007). Non-ruminant porcine fats have very low abundances of C_{44}, C_{46}, and C_{48}, whereas modern milk residues show a wide distribution of TAGs, ranging between C_{28} and C_{54} (Mirabaud et al., 2007; Regert, 2011). Within the archaeological record, shorter TAGs are more vulnerable to hydrolysis degradation, so the distribution of TAGs in degraded ruminant adipose and dairy residues more commonly range between C_{40} and C_{54} (Mirabaud et al., 2007). Due to degradation, the

TAG distribution of archaeological samples is used with caution, and was not further interpreted here. For more reliable information on animal fat contents, stable carbon isotope compositions of individual fatty acids were carried out.

Wax esters occurred in nine out of 13 Mzonjani samples and consisted mainly of palmitate and stearate wax esters with 34–48 carbon atoms, consistent with a plant origin (Hargrove et al., 2004). No indication for beeswax (Regert et al., 2001) or insect waxes (Parish et al., 2008) was identified.

In general, the samples from LHS yielded low concentrations of alcohols. The alcohol distribution within the Mzonjani samples ranged from C_{11}–C_{32}. Alcohols mainly originate as constituents of wax esters and ether lipids. Hence, the alcohols in the LHS samples likely originate from wax ester degradation (Gunstone, 2004) and the biosynthetic pathways of plant ketones (Gašo-Sokač et al., 2014; Raven et al., 1997) as well as from naturally occurring aliphatic alcohols in nature, such as primary and secondary alcohols which are commonly found in plant cuticular waxes (Evershed, 1992).

The distribution of *n*-alkanes within the Mzonjani samples was variable, ranging from C_{11}–C_{35} with a dominance of odd-over-even carbon numbers in most samples, likely originating from various sources, such as bacteria, fungi, algae, higher plants and higher animals. Most Mzonjani samples seem to contain *n*-alkanes originating from plant waxes, with chain lengths between C_{21} and C_{35}, with usually C_{29} and C_{31} being the most abundant (Gunstone and Harwood, 2007).

A total of five samples from the Mzonjani facies contained ketones, mostly representing asymmetrical ones, however, the abundances of ketones from the archaeological samples were too low to estimate the general ketone distribution. Nonetheless, formation during heating, cooking, or post-firing treatments is likely in most cases. However, sample LHS-18 InL2 showed great variety within the ketone distribution, providing a pattern of 1:4:3:1 (K29:K31:K33:K35) (S1, Fig. S17) with smaller abundances of ketones C_{27}, C_{28}, C_{30}, C_{34}), whereas a typical monomodal distribution for ketones deriving from the impact of heat follows a 1:2:1 (C_{31}:C_{33}:C_{35}) pattern (Raven et al., 1997). As C_{31} (16-one) is a symmetrical ketone and relatively more abundant than the other ketones, it seemed possible that some ketones within this sample were of plant origin, such as the symmetrical C_{27} (14-one), due to the presence of ketones in epicuticular leaf waxes (Raven et al., 1997). Raven et al. suggests that the symmetrical C_{35} (18-one) rarely occurs in modern plants (Raven et al., 1997), however, too few organic residue studies have focused on sub-Saharan Africa and its plant world. Hence, the presence of certain ketones and their relative abundances found in South African samples could rather represent the processing of plants instead of the formation of ketones via decarboxylation reactions.

Within the Mzonjani samples, the most common sterol was cholesterol. In addition, sitosterol, stigmastanol, campesterol, and coprostanol were detected in some samples, whereas coprostanol was often found in association with stigmastanol. Sterols and sterol derivatives allow clear distinctions between plant and animal lipid origin and are less likely to be affected by degradation than fatty acids, but may undergo oxidation and reduction reactions (Evershed, 1993). Cholesterol is the most abundant sterol in animal fats, whereas campesterol and β-sitosterol are present in plants (Parish et al., 2008). Cholestan-3-ol (3β,5β) forms from microbial reduction of cholesterol within the mammalian gastrointestinal tract. It is hence used as a biomarker of faecal inputs (Bethell et al., 1994; Bull et al., 2002; Evershed, 1993). The detection of cholesterol is in general interpreted with caution as it could originate from modern contamination, such as vessel handling or leaching from the soil into the ceramic matrix (Evershed, 1993; Hammann et al., 2018). In the case of the Mzonjani samples, the coprostanol, cholesterol as well as the plant sterols and stanols were exclusively found in vessels with a significant lipid concentration and no squalene could be identified in any of the samples which is a common vessel handling contaminant (Evershed, 1993). Furthermore, coprostanol was not detected in any of the soil samples. Hence, the sterols and their derivatives likely derive from archaeological pottery use.

Isovanillin/vanillin occurred in Mzonjani vessels from pits 9 and 10. Most samples yielding the compound isovanillin/vanillin further contained acetovanillone, vanillic acid, 4-hydroxybenzaldehyde and 4-hydroxybenzoic acid (S1, Table S6). Vanillin can naturally derive from certain orchid pods or form as a degradation product of woody tissue, such as lignin (Awan et al., 2019; Huesgen, 2011; Linares et al., 2019). At the current state of research, it can not be ruled out that the vanillin originates from lignin, however, no further degradation markers of lignin were detected. Further investigation will be required to identify the possible source.

3.2. Doornkop facies (Kalundu Tradition)

The Doornkop samples also provided a wide range of compounds representing different mixtures of resources within the same vessel (Fig. 5; S1, Figs. S18–S19; S2, Table S2).

In comparison to the Mzonjani samples, the Doornkop samples provided a wider range of saturated fatty acids, ranging from $C_{9:0}$-$C_{32:0}$, including $C_{16:1}$, $C_{18:1}$, and $C_{19:1}$, as well as branched *iso-* and *anteiso*-fatty acids of $C_{13:0}$, $C_{15:0}$ and $C_{17:0}$. In all samples having a significant quantity of lipid, $C_{16:0}$ and $C_{18:0}$ were the most abundant of all fatty acids. Similar to the Mzonjani samples, the fatty acid distribution can originate both from plant and animal fat (Regert, 2011). Two Doornkop vessels contained ricinoleic acid (LHS-24 and LHS-36-37). Although the degradation marker 9,12-dihydroxyoctadecanoic acid was not detected, a modern contamination seems highly unlikely. Ricinoleic acid was not detected in any of the soil controls nor in any other samples, hence contamination from the burial environment can be excluded. Possible sources for ricinoleic acid include ergot fungi (genus *Claviceps*) and *Ricinus communis*. Other local plant species whose lipid composition has not yet been studied might also prove possible sources.

Nine of 13 Doornkop samples yielded evidence for acylglycerols. The most common TAGs were even-numbered molecules ranging between C_{46} and C_{52} and the correlated degradation products could be observed within the DAGs' and MAGs' profile. The distribution of TAGs in the Doornkop sherds was wider than in the Mzonjani sherds. Despite the wider range, the TAG profile is too degraded to draw any detailed conclusions regarding the animal or plant source (Mirabaud et al., 2007).

Wax esters occurred in six of the 13 Doornkop samples. As in the Mzonjani samples, the wax esters consist mainly of palmitate and stearate wax esters comprising C_{35}–C_{48} carbon atoms consistent with a plant origin (Hargrove et al., 2004). Again, no indication for beeswax (Regert et al., 2001) or insect waxes (Parish et al., 2008) was identified.

The alcohol distribution within the Doornkop samples ranged from C_{12}–C_{32}. As is the case with the Mzonjani samples, the alcohols mainly originate from wax esters and plant ketones (Gunstone and Harwood, 2007; Gurr et al., 2002).

The distribution of *n*-alkanes within the Doornkop samples was variable, ranging from C_{11}–C_{32} with a dominance of odd-over-even carbon numbers in most samples, likely originating from various sources, such as bacteria, fungi, higher plants, and higher animals (Gunstone and Harwood, 2007).

Four of the 13 Doornkop samples contained asymmetrical ketones ranging from C_{29} to C_{35}, whereas sample LHS-13 InL2 also contained C_{34}. Only sample LHS-28 InL2 shows the typical distribution of 1:2:1 (K31:K33:K35) of ketones (S1, Figs. S18 and S19), possibly deriving from the impact of heat. All other Doornkop samples yielded only traces of ketones with abundances too low to investigate their distribution. Their origin could be variable, such as heat impact and plant input (Drieu et al., 2020; Evershed et al., 1995; Raven et al., 1997), and can not be investigated further at this stage.

As with the Mzonjani samples, the most common sterol was cholesterol, followed by sitosterol, stigmastanol, campesterol, and coprostanol, with the latter often found also in association with stigmastanol.

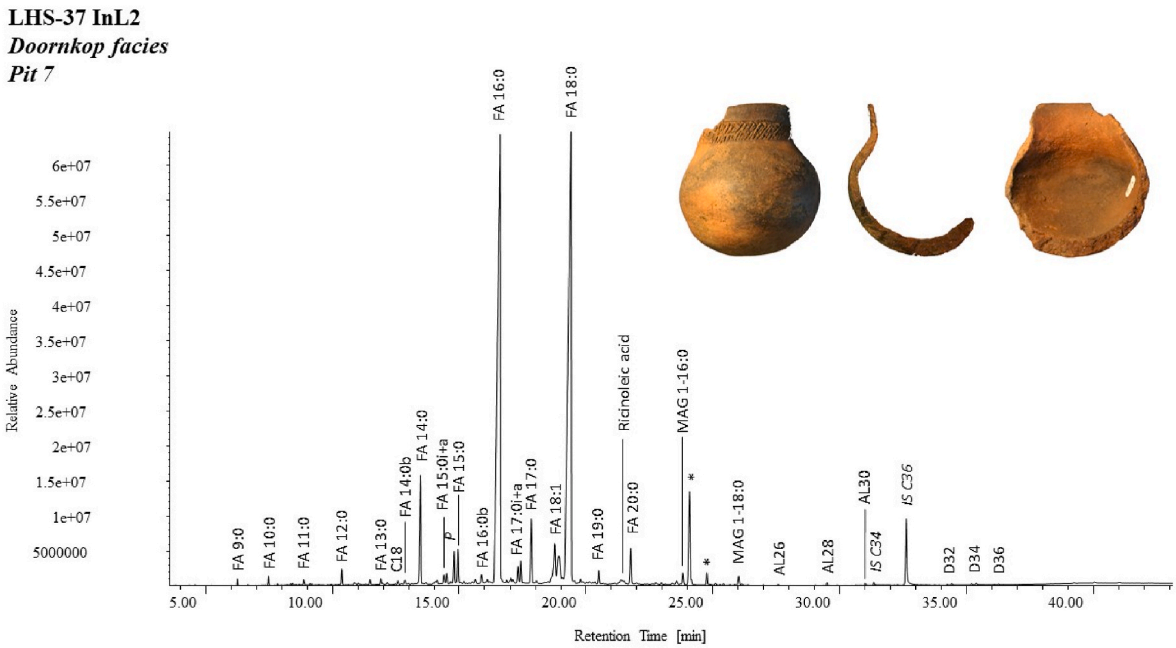

Fig. 5. Total Ion Chromatogram (TIC) of sample LHS-37 InL2 from the Doornkop facies, LHS, containing ricinoleic acid. FAx:y: Fatty acids showing the number of carbon atoms (x) and the degree of unsaturation (y); C: Alkanes; AL: Alcohols; WE: Wax esters; MAGx-y:z: Monoacylglycerols showing positioning on glyceride (x), the number of carbon atoms (y) and the degree of unsaturation (z); D: Diacylglycerols; T: Triacylglycerols; P: Phthalate; IS: Internal Standards.

The plant sterols and stanols as well as coprostanol and cholesterol were exclusively found in Doornkop vessels with a significant lipid concentration. Furthermore, squalene was not detected in any of the Doornkop samples. Hence, an archaeological origin for the sterols and their derivatives seems highly likely, probably originating from animal and plant sources (Evershed, 1993; Parish et al., 2008).

Some samples from pits 5, 7, and 11 of the Doornkop facies contained the compounds isovanillin/vanillin, acetovanillone, vanillic acid, 4-hydroxybenzaldehyde and 4-hydroxybenzoic acid (S1, Table S7). As described in the Mzonjani results section, these compounds can naturally derive from certain orchid pods or form as a degradation product of woody tissue, such as lignin (Awan et al., 2019; Huesgen, 2011; Linares et al., 2019). The origin of these molecules needs further investigation.

4. Compound-specific isotope evidence (Gas Chromatography-Combustion-Isotope Ratio Mass Spectrometry)

A total of 19 archaeological samples (Mzonjani $n = 9$; Doornkop $n = 10$) and one modern reference sample (Common duiker) were submitted for compound-specific isotope analysis. The GC-C-IRMS results of the LHS samples ranged in their $\delta^{13}C_{16:0}$ values from −21‰ to −14‰, with samples from the Doornkop facies tending to be slightly less negative (−19‰ to −14‰; average of −15.89‰) in comparison to the Mzonjani facies (−21‰ to −16‰; average of −18.02‰). The Δ^{13}C-values ranged in the Mzonjani samples from −4.62 to 0.84 and in the Doornkop samples from −3.83 to −0.64 which is consistent with modern references from non-ruminant and ruminant adipose fats as well as ruminant dairy (Fig. 6; S1, Tables S4–S5). The majority of samples from the LHS plot within the range of ruminant adipose fat ($n = 11$; 63%), followed by dairy fats ($n = 6$; 32%). Samples LHS-36-37 originate from the same vessel. Sample LHS-10 falls just outside the $\pm$ 1σ standard deviation of the ruminant milk range, possibly representing a mixture of ruminant adipose and milk fats. Within the Mzonjani facies, only one sample, LHS-27, plots in the non-ruminant adipose range ($n = 1$; 5%).

As no modern reference database from sub-Saharan Africa is available, results are compared using the globally applicable Δ^{13}C-values ($\delta^{13}C_{18:0}$ – $\delta^{13}C_{16:0}$) plotted against $\delta^{13}C_{16:0}$. This allows good comparison of lipids which derive from either mixed C_3 and C_4 or pure C_3/C_4 sources (Dunne et al., 2012). In general, the δ^{13}C values of fatty acids reflect the animal diet, including the C_3 and C_4 composition of their respective diets, hence the biosynthetic pathway within organisms (Stott et al., 1997). By using the Δ^{13}C-values, the environmental effects on δ^{13}C variation and the metabolic and physiological variations between animals are excluded (Evershed et al., 2002). The LHS Δ^{13}C-plot is based on published reference data from Libya and Kenya (Dunne et al., 2012), UK (Dudd, 1999; Dudd and Evershed, 1998), Kazakhstan (Outram et al., 2009), Switzerland (Spangenberg et al., 2006) and the Near East (Gregg et al., 2009).

In comparison to Central and Northern European references (see e.g. Copley et al., 2005a, 2005b, 2005c, 2005d, 2003; Mukherjee et al., 2008, 2007; Salque et al., 2013), the $\delta^{13}C_{16:0}$ of the LHS samples is wider due to the mixed C_3 and C_4 plant contribution to the diet. The broadening of values based on the C_4 plant contribution can also be observed outside Africa in certain time periods from e.g. Southeastern Europe and the Levant (see e.g. Evershed et al., 2008; Gregg et al., 2009; Hendy et al., 2018; Spiteri, 2012). Few available studies on compound-specific isotope analyses from Saharan to sub-Saharan Africa provided an even wider range (−30‰ to −10‰) than the one observed in Europe (−30‰ to −25‰) and the Levant (−30‰ to −20‰), however, often with clusters between −20‰ and −10‰ (Copley et al., 2004; Dunne et al., 2012, 2019; Fewlass et al., 2020; Grillo et al., 2020; Hopper et al., 2023; Roffet-Salque et al., 2017b). Hence, the larger range within the LHS samples can be explained by the contribution of C_4 plants to the animals' diet.

5. Discussion

The lipid preservation within the LHS pottery is exceptional. Various factors may have influenced this, including climatic conditions, soil chemistry (Aillaud, 2002; Eglinton and Logan, 1991; Moucawi et al., 1981), and differences in pottery use and ceramic fabric (Roffet-Salque et al., 2017a; Spiteri et al., 2017). There seems to be great variation in terms of lipid concentration obtained from the ceramic samples. As the sherds originate from the same area, pits or even spits, no distinct environmental advantage or disadvantage could be identified leading to differences in lipid preservation. It is therefore highly probable that the

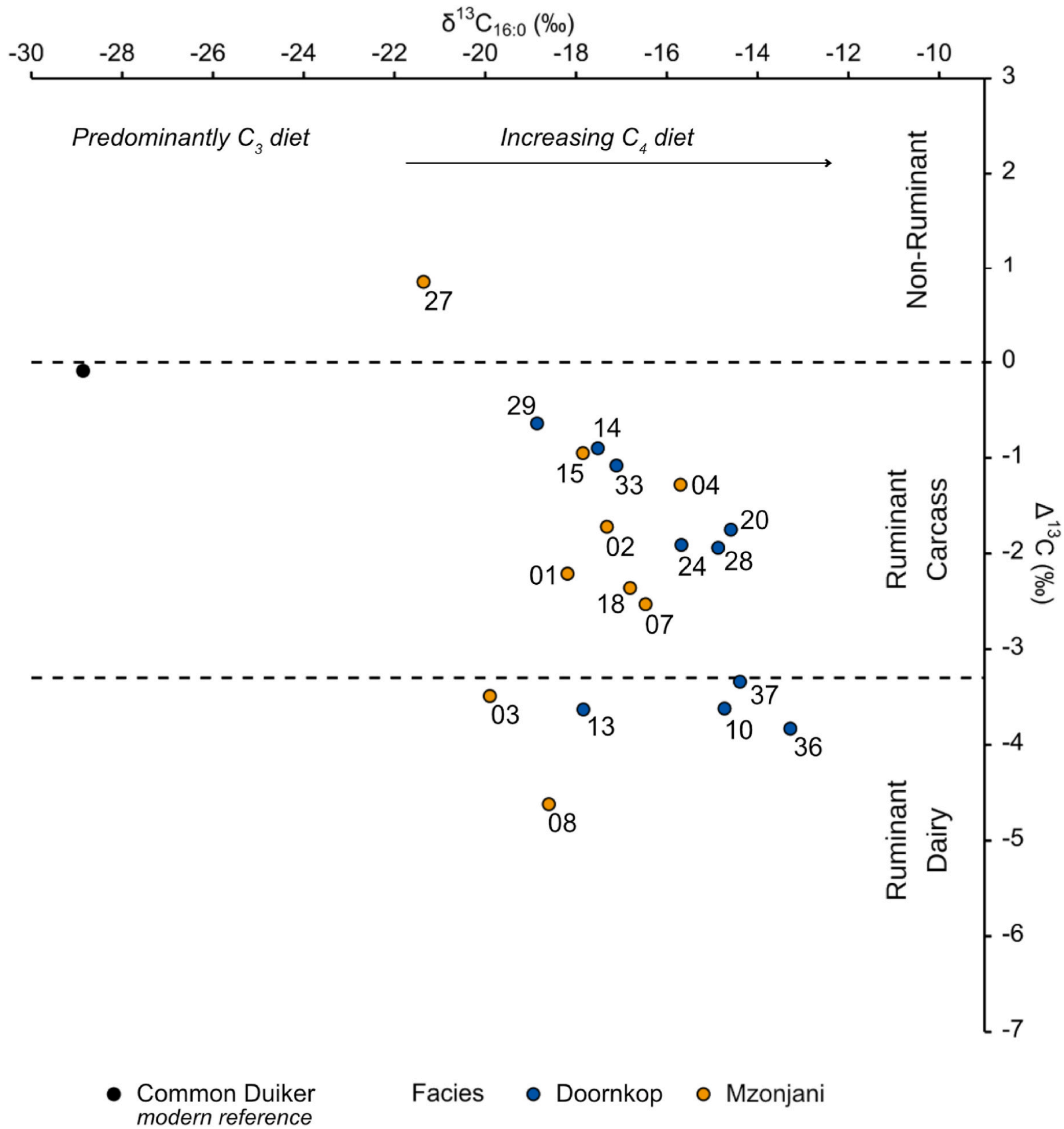

Fig. 6. GC-C-IRMS results of the Mzonjani (in orange) and Doornkop (in blue) facies samples, data points labelled after sample number. Modern reference sample of common duiker in black (S1, Table S8); $\Delta^{13}C$ plot against $\delta^{13}C_{16:0}$ values; $\pm$ 1σ standard deviations of non-ruminant and ruminant adiposes, and ruminant dairy plotted after published reference data from Africa (Dunne et al., 2012), UK (Dudd, 1999; Dudd and Evershed, 1998), Kazakhstan (Outram et al., 2009), Switzerland (Spangenberg et al., 2006) and the Near East (Gregg et al., 2009); modern references corrected by −1.14 to correct for post-Industrial Revolution effects of fossil burning and resulting change in atmospheric CO_2 (Friedli et al., 1986). (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

samples are directly comparable in terms of use and hence lipid concentrations. Via the application of the conventional lipid extraction method, half of the ceramic samples yielded significant amounts of lipids. The LHS samples are currently under further investigation using an acidified methanol extraction method to examine the vessels that have shown negligible residue, involving further compound-specific isotope analysis and undertaking a general plant and fish marker screening applied to all samples to check for the presence of long-chain ω-(o-alkylphenyl)alkanoic acids (APAAs).

The obtained ORA data from LHS confirms the presence of ruminant and non-ruminant carcass fats as well as ruminant dairy fats. It is hence likely that most ruminant adipose samples represent the processing of *Ovis/Capra* species and domestic cattle (*Bos taurus*), with possible evidence of yet unknown wild ruminants among the bovid size classes I-III, such as antelopes.

Two Mzonjani (LHS-08, LHS-03) and three of the Doornkop samples (LHS-13, LHS-36-37), each representing two vessels, plot within the ± 1σ standard deviation of ruminant dairy. It is most probable that the obtained dairy products derive from domestic species, such as sheep, goat or cattle. It has already been demonstrated elsewhere that dairy products are of significant importance to pastoralist communities in eastern and southern Africa (e.g. Dunne et al., 2019; Fewlass et al., 2020). This would support the suggestion that ruminant meat and dairy products contributed to the diet of early farmers (e.g. Voigt and Plug, 1984), although dairy products from small stock were probably of minor importance (Badenhorst, 2018). In the southern African farmer context, secondary products such as sour milk, curds and whey, or butter, should be considered especially important, as is the case with early farming communities in Europe (Craig et al., 2005). Bantu-speaking people generally display a low percentage of lactase persistence (Breton et al., 2014; Torniainen et al., 2009) and sour, clotted milk was much preferred over fresh milk. Where fresh milk was consumed, it was commonly boiled; young boys also drank directly from the udder while milking. As noted earlier, various farming communities in South Africa relied (and still rely) on daily consumption of sour milk (known in Zulu as *amasi*), often mixed with various vegetables (Krige, 1962, p. 55; Quin, 1959, pp. 226–227; Sansom, 1974). Similar practices are known from Lesotho, Botswana, and Zimbabwe (Gadaga et al., 2013). Through the consumption of the traditional fermented buttermilk, which is low in lactose content, southern African farmers are able to consume larger amounts of dairy products (de Vrese et al., 2001). The identification of dairy products in both occupation facies at LHS so far appears to represent the first direct evidence for the processing of dairy products by early farming communities in southern Africa.

All dairy samples, except LHS-08, plot close to the higher 1σ standard deviation range of reference ruminant milk. All ruminant carcass and dairy samples, except for LHS-28, also provided strong evidence for the processing of plants comprising long-chain fatty acids, alkanes and alcohols, as well as sitosterol, campesterol, stigmastanol, and many different mixtures of mostly palmitate and stearate wax esters. These distributions might indicate the processing of leafy vegetables (e.g. Eglinton and Hamilton, 1967; Evershed et al., 1991), seed oils (Soomro and Sherazi, 2013) or as yet unknown plants (Drieu et al., 2018; Ribechini et al., 2008). In South Africa, almost all savanna and grassland grasses, as well as domestic crops, are C_4 plants, whereas nearly all gathered wild food sources, such as tubers, fruits, and cultivated legumes and cucurbits, cluster under C_3 plants (Lee-Thorp et al., 1993). The theoretical mixing model put forward by Hendy et al. (2018), suggests that Δ^{13}C- values of ruminant adipose and milk fats can be altered due to the input of plant lipids. Hence, potentially, the mixing of milk from animals with variable C_3 and C_4 diets and C_3 plant lipids can result in more positive Δ^{13}C-values, although this hypothesis still needs to be proven by carrying out cooking experiments. It is therefore possible that some of the isotopic values from the LHS represent mixtures of animal fats and leafy vegetables which have been observed in the ethnographic record (Quin, 1959, pp. 61–80).

The Δ^{13}C against $\delta^{13}C_{16:0}$-plot can reveal differences in animal diets, especially with regard to C_3 and C_4 plant contribution to the diet (Dunne et al., 2012). Between the two facies at LHS, a shift towards more positive $\delta^{13}C_{16:0}$ values was observed (see Fig. 6) which might be explained by several scenarios. (1) These variations might be linked to natural differences in feeding habits of animals with distinct diets. The diet of browsers (e.g. goats) and grazers (e.g. cows/sheep) can vary significantly: browsers mostly feed on C_3 plants, grazers mainly consume C_4 plants. Consequently, browsers should produce more negative values in comparison to grazers, whereas samples plotting in between could represent mixtures of processed fats. Climatic reconstructions of the Lydenburg area show relatively stable, with moderate to dry conditions, and a dominance of C_4 related grasses (Chloridoideae, Panicoideae) in relation to C_3 (Pooideae) grasses from 400 CE–1000 (Sjöström et al., 2017). This would imply that the environment during the Mzonjani and Doornkop occupation was comparable, hence, the shift in the isotopic data could be explained by the processing of different species (shift from e.g. goats to more cow/sheep). (2) Another possibility might be differences in animal husbandry practices, such as foddering. This would result in a human-induced shift which can be observed in the publication by Fewlass et al. (2020) where the impact of maize foddering (C_4-plant) in the late 19th and 20th centuries could be observed. As maize was only introduced later in the 2nd millennium CE with the arrival of Europeans, the possible available domestic plants for foddering were sorghum and millet (both C_4-plants). (3) Some of these samples might represent the processing of wild ruminant species. A study conducted by Craig et al. (2012) on European deer demonstrated that wild ruminants can plot below −3.3‰ within the Δ^{13}C-plot. As no modern references from wild species, especially wild ruminants from sub-Saharan Africa are available, it remains a possibility that some of the samples may represent wild ruminant processing instead of dairy. The reasons for shifts to more positive $\delta^{13}C_{16:0}$ values within the LHS samples remain uncertain for now. The sample size was relatively small, hence, secure conclusions could not be drawn and the three different scenarios remain working hypotheses. In the future, the sample size needs to be expanded to confirm these observations in combination with modern reference samples from the region.

During the first millennium CE, a wide range of wild animals were exploited. Within the Mzonjani facies, remains of the Common duiker (*Sylvicapra grimmia*) were identified. Hence, a modern reference sample was submitted for compound-specific isotope analysis (see Fig. 6). Based on the isotopic values, the duiker could be excluded as a wild ruminant source for any of the LHS samples. This is due to the animals' omnivorous feeding habits, which results in different C values and do not match with the archaeological samples.

Within the Mzonjani facies, a sample provided evidence for the processing of non-ruminant adipose fat (LHS-27). In the European context, the non-ruminant range is often interpreted as of porcine origin, due to the presence of domestic pig (see e.g. Mukherjee et al., 2008, 2007). *Sus scrofa domesticus*, however, was introduced to South Africa by European Settlers only from the 17th century CE onwards (Epstein, 1971; Gifford-Gonzalez and Hanotte, 2013). Hence, the non-ruminant adipose sample possibly represents a wild non-ruminant species. Wild pig species, such as warthog (*Phacochoerus aethiopicus*) and bushpig (*Potamochoerus larvatus*) are commonly identified in early farming faunal assemblages (Smith, 2000), however at LHS, no faunal remains of wild pig were found possibly due to the nature of the deposition (see S1, Tables S1 and S2). Other possibilities for the exploitation of wild non-ruminants include hyrax (*Procavia* spp.), which was identified in the faunal assemblage of the younger Doornkop facies, hare (*Lepus* spp.), zebra (*Equus* spp.), elephant (*Loxodonta africana*), aquatic sources, guinea fowl, and various tortoise, or even monkey species, just to name a few. The domestic chicken (*Gallus domesticus*) is another possibility. However, neither the early farming context of LHS nor other sites dating to the same time period in the area provided evidence for domestic chicken. In South Africa, the earliest identification for the presence of domestic birds dates to the 7-9th century CE, identified at KwaGandaganda (Msuluzi phase) (Beukes, 2000; Whitelaw, 1994) and 9-10th century CE, identified at Ndondondwane, KwaZulu-Natal (Clutton-Brock, 1993; Plug and Voigt, 1985; Voigt and Von den Driesch, 1984), which both post-date the Mzonjani facies at LHS. At the current stage of research, the origin of the non-ruminant fat cannot be examined in further detail.

When comparing the vessel profiles of the LHS sherds with the identified lipid contents in this study, no correlation could be observed as suggested by ethnographic observation (Fowler, 2006). Consequently, ethnographic studies, which focus on the correlation of use and typology e.g. (Fowler, 2006; Huffman, 1972), should be applied with caution to EIA pottery. However, there seems to be a tendency for ruminant processing in vessels with decoration motif modes D1, D3, and D5 during the Doornkop occupation. In general, larger datasets from lipid residue analysis, especially GC-C-IRMS data, are needed to better evaluate correlations between vessel shape, size, decoration and the use of ceramic vessels. Ethnographic observations by e.g. Fowler (2006) and Huffman (1972) could be used to test against the evidence provided by early farming pottery analysis, helping to verify observed patterns or to even discover yet unknown ones. However, it is also important to note that from first contact with the colonial world, imported vessels replaced some local vessels, hence, the modern sets used for some ethnographic work are reduced versions of what once existed (Fowler, 2006; Hall, 1997). Although function largely is still related to shape and size, ORA could be a helpful tool to explore these relations in the past.

Interestingly, no biomarkers representing the use of beeswax/honey were detected in any of the LHS samples, although they have been sporadically identified at early farming sites elsewhere in Africa via organic residue analysis (Crowther et al., 2015; Dunne et al., 2021). Especially in southern Africa, only small amounts of sugar could be gathered from plants, whereas honey would have offered access to high amounts of sugars within short periods of time (Russell and Lander, 2015). Most ORA studies applied to sub-Saharan sites however do not report on the detection of beeswax related compounds, except the study by Dunne and colleagues, who investigated the early farming Nok culture from West Africa (Dunne et al., 2021). The importance of beeswax/honey for early farming societies in southern Africa must remain an open question for now until more ORA studies are available.

Ricinoleic acid occurred exclusively in two Doornkop vessels (LHS-24, -36-37) dating between the 9th and 10th century CE. Generally, the origin of ricinoleic acid is often associated with *Ricinus communis*, and hence castor oil (Copley et al., 2005e). In antiquity, castor oil was used as an illuminant (Colombini et al., 2005; Copley et al., 2005e), as an ingredient in embalming products in ancient Egypt (Rageot et al., 2023; Tchapla et al., 2004), and in medicine for skin and as purgatives (Pecci et al., 2010). Although *R. communis* is not believed indigenous to southern Africa, seeds have been found sporadically in southern African archaeological contexts that date from 3100 BP, but especially those of the 2nd millennium CE (Deacon, 1986; Liengme, 1987; Wadley, 1979; Whitelaw and Mazel, in press). The archaeological seeds have not been directly dated, however, and it remains uncertain whether the seeds are truly associated with the charcoal dates or if they moved through the deposits (Deacon, 1986). In the ethnographic record of Sotho and Zulu traditional medicine, the seeds are sometimes used as purgatives, stomach aches are treated with leaf infusions and the roots and leaf poultices are applied to wounds, sores, and boils (van Wyk et al., 2017).

Ricinoleic acid has also been identified on a wooden stick, interpreted as a poison applicator and dated to 24,000 BP, at Border Cave (d'Errico et al., 2012). d'Errico et al. argue that it derives from *R. communis*. Bradfield et al. (2015) suggest the residue might instead derive from the toxin abrin from the indigenous South African plant *Abrus precatorius* (crab's eye creeper). Whatever the case with the Border Cave residue, little research has been conducted in the African context on possible sources of the ricinoleic acid other than *R. communis*. This compound can also occur in *Jatropha* spp. (Euphorbiaceae), a genus which is found throughout tropical and subtropical regions in the world (Hernández-Nicolás et al., 2018). Several species of *Jatropha* (*J.*) are native to southern Africa: *J. capensis*, *J. erythropoda*, *J. hirsuta*, and *J. zeyheri* (Pooley, 1998; van Wyk et al., 2017; Viswanathan et al., 2018; Welcome and Van Wyk, 2019). *J. hirsuta* is a source of traditional medicine for treating fever and wounds, as well as being a protective charm against lightning (Pooley, 1998). Several other species have recognized medicinal properties (van Wyk et al., 2017; Welcome and Van Wyk, 2019), but the lipid profiles from various plant parts are yet to be investigated. Mongalo et al. (2019) investigated the medicinal aspects of *J. zeyheri* roots, but found no evidence for ricinoleic acid. This finding, however, does not exclude either other parts of the *J. zeyheri* plant or other southern African *Jatropha* spp. that contain ricinoleic acid, such as *J. hirsuta* or seasonal variation of ricinoleic acid within any of the *Jatropha* species.

Potters throughout Africa treat pots with various organic materials to strengthen, waterproof or finish them. In some groups, potters rub the surfaces of fired pots with the leaves of plants that can be both indigenous and exotic in origin (Armstrong et al., 2008, p. 524; Gosselain, 2016). Fon potters in Benin use the leaves of *Ricinus communis* for this purpose (Gosselain, 2002, p. 190). At LHS, ricinoleic acid was not detected in the exterior vessel sample of LHS-24 and was present in vessel LHS-36-37 in much lower abundances in the base (LHS-36) in comparison to the shoulder sample (LHS-37). The latter pattern correlates with the typical lipid distribution pattern for boiling processes (Charters et al., 1997). Hence, a post-firing treatment of leaf-rubbing seems unlikely, as the compound should then be detectable throughout the interior, and possibly also exterior, of the ceramic matrix. The boiling of plant material containing ricinoleic acid can be either interpreted as evidence for medicinal purposes or post-firing treatment to strengthen or waterproof the vessel. In case of a post-firing treatment tradition, a higher number of pottery samples would be expected to contain ricinoleic acid and it is rather unlikely that once-off leaf rubbing of a fired pot would leave much of a residue. It cannot be excluded that perhaps at LHS, *Ricinus communis*, *Jatropha* spp., or another unidentified species was utilised for medicinal purposes during the Doornkop occupation facies.

Coprostanol is commonly used as a biomarker for investigating soil chemistry (Bethell et al., 1994; Prost et al., 2017), mummies (Gülaçar et al., 1990), bog bodies (Evershed, 1990; Evershed and Connolly, 1994), and coprolites (Lin et al., 1978). As coprostanol/cholestan-3-ol (3β,5β) is a product of the microbial reduction of cholesterol, which is produced in the mammalian gastrointestinal tract, it can be used as a biomarker for faecal inputs (Bethell et al., 1994; Bull et al., 2002; Evershed 1993). In the case of LHS, several possibilities could explain the presence of coprostanol in the archaeological samples. Based on the African ethnographic record, various applications for the combined use of pottery and animal manure are documented. Dung is used during the clay process, firing and post-firing treatments, but also for supernatural reasons, such as smearing very large pots with dung in order to prevent lightning from striking the house (Lawton 1965, p. 52). All samples containing β-coprostanol, except LHS-28, were found together with 5β-stigmastanol which is dominant in ruminant faeces (Bethell et al., 1994; Bull et al., 2002). Thus, the combined presence of coprostanol and stigmastanol might indicate the use of ruminant dung. This is supported by the ethnographic record, providing evidence for the use of livestock dung in ceramic manufacture (Gosselain 2016). For now, there are several working hypotheses which could explain the presence of coprostanol within the ceramic matrix. The ethnographic evidence shows that dung was frequently used to seal vessel lids and cover pots in grain storage pits (Hammond-Tooke, 1993, p. 206; Lawton, 1965, pp. 320, 333). Due to the further presence of degraded animal fats within the LHS pots, it is evident that the vessels were not used exclusively for grain storage. Another possibility known from the ethnographic record implies the smearing of wet cow dung onto the ceramic surfaces during post-firing treatments to strengthen and waterproof the body (Gosselain, 2016, pp. 1086–1087, 2002, p. 191; Lawton, 1965, p. 144). Vessels must have been treated with dung not only once after manufacture, but multiple times within the vessels' lifespan, when also used as cooking vessels. It has also been demonstrated by Pecci et al. that sealants tend to wear-out over time and need to be renewed (Pecci et al., 2016). A third possibility remains the mending of pottery with dung to fill minor cracks (Lawton, 1965, p. 423). Cracked pottery is not ideal to use for cooking, hence, the pots might have been repaired with dung and recycled for a different purpose. At the current stage of the study, the record from LHS implies that dung could have been involved in different forms of pottery sealing and/or mending during both occupation facies. For now, the origin of coprostanol within the LHS samples cannot be further distinguished but further investigations are being carried out to explore the source of coprostanol, which include the investigation of stanol distribution pattern and other biomarker molecules, such as bile acids (Bethell et al., 1994; Harrault et al., 2019; Prost et al., 2017).

The compound vanillin/isovanillin was often found in association with acetovanillone, vanillic acid, 4-hydroxybenzaldehyde and 4-hydroxybenzoic acid. These compounds can naturally derive from certain orchid pods or form as a degradation product of woody tissue, such as lignin (Awan et al., 2019; Linares et al., 2019). Based on the distribution of compounds throughout samples and pits, there is a possibility that they originated from lignin. No typical pattern of lignin degradation markers, however, was detected, even though there are over 50 biomarkers formed through the decomposition of lignin (Faix et al., 1990; Javor et al., 2000). None of these compounds was detected in the soil control samples. Hence, other sources, such as orchid species, are a possible source. In southern Africa, there are approximately 494 indigenous terrestrial and epiphytic orchids, 49 of which are used in traditional African medicine (Chinsamy et al., 2011) and 11 species used as food (Welcome and Van Wyk, 2019). Furthermore, isovanillin was detected in roots of the Medicinal African Liane, *Mondia whitei*, which is a popular ethnomedicine to treat various medical conditions (Koorbanally et al., 2000). In order to determine the possible source for these markers, investigations of modern references of indigenous vanilla species and their lipid profiles are needed. This will help to examine whether early farmers processed vanilla containing orchid pods for possible flavouring or medicinal purposes.

6. Conclusion

Despite the intense research on early farming societies in southern Africa, conducted over the past decades, little is known about the use, content or purpose of certain vessel types. Our study demonstrates that ORA is a useful method to test assumptions based on ethno-archaeological and typological approaches. LHS provided the earliest evidence for the processing of dairy products by early farmers in southern Africa. Due to a high percentage of Bantu-speaking people with a lactase deficiency, it is highly likely that the dairy evidence represents secondary milk products, such as sour milk and curds of milk, rather than the consumption of fresh milk. Furthermore, the obtained ORA data from LHS confirms the processing of ruminant carcass fats within both occupation facies, likely representing domestic animal fats, as well as the processing of wild non-ruminant carcass fats within the earlier Mzonjani facies. In addition, we identified the use of a ricinoleic acid containing plant which may point towards medicinal purposes. This implies that ethnographically documented practices might reach back to the 1st millennium CE. In both occupational facies, it remains a possibility that early farmers processed vanilla containing orchid pods for possible flavouring or medicinal purposes.

The evidence from LHS suggests mixing animal fats to plant oils, and that no distinction in pot use was made in both occupation facies, a pattern that is commonly identified from ORA studies. However, there might be a tendency towards ruminant processing within the Doornkop vessels which correlates with certain decoration styles. This observed pattern needs further investigation within future ORA studies. In addition, the record from LHS implies that dung could have been involved in different forms of pottery sealing and/or mending during both occupation facies.

The results of this study shed light on how ORA can be an extremely powerful tool for investigating various aspects of vessel use, generating complementary data in the exploitation of various sources and greatly enhancing our understanding of early farming subsistence and establishing a good foundation for future research in this area.

Author contributions

JB, CS and AS acquired funding. JB, CS, AS and GW designed the study. JB, AS and J-PC provided samples. JB, CS, SB, SC, MB and MR analysed the data. JB, CS, AS and GW interpreted the data. JB, CS, AS and GW drafted the main manuscript and JB prepared figures. All authors reviewed the manuscript.

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Publisher's note

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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