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Residue Analysis of Middle Bronze Age Vessels from the Burial Cave at Beit Ṣafafa, Jerusalem

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Introduction

Approximately 65 pottery vessels from Middle Bronze Age II burial Cave I at Beit Ṣafafa (see Ben-Ari and Wiegmann, this volume) were found; 55 of them were sampled and submitted to Absorbed Organic Residues screening and analyses. Each vessel subjected to analysis was sampled prior to washing. Forty-nine results are presented in Table 1.

The cave was found partially destroyed and had been exposed to the effects of the environment. Most of the excavated artifacts were discovered crushed, damaged, crumbled and in an extreme stage of disintegration. Therefore, it is no surprise that almost half the extracts (26 of the 55 analyzed items) showed a "typical contamination" molecular assemblage. All the extracts contained less than 10 µg of lipids per one gram of ceramic powder. Nevertheless, in all other specimens where unique identifiable molecular assemblages were detected, the amount of organic compounds exceeded this amount, thereby substantiating that the origin of these compounds was genuine and not contaminated.

Methods

Extraction of Organic Residues

All laboratory glassware was soaked overnight in fuming nitric acid, washed carefully with distilled water, and then washed with acetone, followed by dichloromethane and dried in a fume hood. Method-blanks were routinely run with each batch of extraction, for both archaeological and modern samples, as well as for plant extraction. This routine was employed to monitor and detect any introduction of contaminants during lab work.

A 1 sq cm fragment was broken off each ceramic vessel with a plier after which the surface was carefully cleaned with a sterile scalpel and then manually ground to a powder in an agate mortar and pestle. One gram of the homogenized powder was used for the extraction. Each sample was placed in a 10 mL glass tube. The following steps were repeated twice: 10 mL of a dichloromethane:methanol mixture (2:1, v/v) was added to each sample, followed by sonication for 10 minutes; the tubes were then centrifuged for 10 minutes at 3500 rpm. The

Table 1. List of Pottery Sampled for Residue Analysis and Their Lipid Content

Location	No.	Locus	Basket	Type	Description	Figure[i]	Comments
Dump	1	99	1061	Dipper juglet	A piece of the body was sampled	6:7	Complete vessel
	2	99	1075	Deep carinated bowl	Sampled close to the base	6:4	Complete vessel
Group 1	3	102	1096	Dipper juglet		10:8	Complete vessel
	4	102	1093/2	Bowl	sampled close to the base		Sherds
	5	102	1000/5	Deep carinated bowl	The base was sampled	10:3	Complete vessel
	6	102	1000/1	Deep carinated bowl	The base was sampled		A disk base
	7	102	1091/6	Lamp bowl	Sampled close to base	10:2	Body of a carinated bowl
	8	102	1002/1	Jug	The base was sampled	10:5	Complete vessel
	9	102	1002/2	Jug	The base was sampled	10:7	Almost complete vessel
	10	111	1085	Black piriform juglet	Sampled close to base		Body sherds and a base only
	11	111	1067	Pithos	A piece of the body was sampled	10:4	Complete vessel
Group 2	12	105	1025	Black piriform juglet	The base was sampled		Body sherds and a base only
	13	105	1020/1	Twin vessel	The base was sampled	15:5	Complete vessel
	14	105	1020/2	Twin vessel	The base was sampled	15:5	Complete vessel
	15	105	1024	Piriform juglet	The base was sampled	15:6	Complete vessel
	16	105	1098/1	Platter bowl	Sampled close to the base	15:2	Complete vessel
	17	105	1098/2	Platter bowl	sampled close to the base	15:3	Complete vessel
	18	105	1022/1	Platter bowl	The base was sampled	15:1	Complete vessel
	19	105	1023	Deep carinated bowl	Sampled close to the rim	15:4	Complete vessel
	20	105	1021	Bowl	Sampled close to the base		Thick disk base

[i] See Ben-Ari and Wiegmann, this volume.

Table 1. (cont.)

Location	No.	Locus	Basket	Type	Description	Figure[i]	Comments
Group 3	21	104	1019	Deep carinated bowl	Sampled close to rim	16:1	Complete vessel
	22	104	1018	Deep carinated bowl	The body was sampled	16:3	Complete vessel
	23	104	1017/1	Deep carinated bowl	The base of bowl 1/2 was sampled	16:2	Complete vessel
	24	104	1017/2	Deep carinated bowl	The base of bowl 1/2 was sampled	16:2	Complete vessel
Group 4	25	106	1040/1	Juglet	The base was sampled		Sherds
	26	106	1040/2	Juglet	The base was sampled		Sherds
	27	106	1042	Deep carinated bowl	The base was sampled	17:2	Complete vessel
	28	106	1044	Deep carinated bowl	The base was sampled	17:1	Non restorable vessel
	29	106	1030	Deep carinated bowl	body was sampled	17:3	Complete vessel
	30	106	1036	Deep carinated bowl	Sampled close to base	17:4	Complete vessel
Group 5	31	103	1008	Black piriform juglet	Sampled close to rim.	19:3	Complete vessel
	32	103	1007/1	Dipper juglet	The base was sampled	19:2	Complete vessel
	33	103	1007/2	Bowl	The base was sampled		Sherds
	34	103	1006	Jar	Sampled close to base	19:1	Complete vessel
Group 6	35	112	1090	Piriform juglet	A piece of the body was sampled	22:9	Complete vessel
	36	112	1083	Dipper juglet	The base was sampled	22:11	Complete vessel
	37	112	1088/1	Piriform juglet	Double handled juglet. Sampled close to base	22:10	Complete vessel

Table 1. (cont.)

Location	No.	Locus	Basket	Type	Description	Figure[i]	Comments
Group 6	38	112	1088/2	Piriform juglet	Double handled juglet. Sampled along the body		
	39	112	1072	Platter bowl	Sampled close to base	22:2	Complete vessel
	40	112	1081	Deep carinated bowl	Sampled close to base		Body sherds and a base
	41	112	1073	Deep carinated bowl	Sampled close to the base.	22:6	Complete vessel
	42	112	1076	Deep carinated bowl	The base was sampled	22:3	Complete vessel
	43	112	1089	Deep carinated bowl	Sampled close to the base		Body sherds and a base
	44	112	1082	Deep carinated bowl	Sampled close to base	22:4	Complete vessel
	45	112	1069	Jar	The base was sampled	23:1	Complete vessel
	46	112	1066	Pithos	The base was sampled	23:3	Complete vessel
	47	112	1068	Pithos	Sampled towards the base	23:2	Complete vessel
	48	112	1064	Bowl on a stand	lower part of bowl was sampled	22:8	Complete vessel
L99	49	99	1077/6	Cooking pot	The body was sampled	6:6	Complete vessel

supernatant was removed to a clean glass vial and 10 µL of 1-nonadecanol (C19ol) diluted in DCM:MeOH was added to serve as an internal standard. The accumulated solvents were evaporated under a gentle stream of nitrogen. Prior to analysis 100 µl of N,O-bis(trimethylsilyl) trifluoroacetamide (BSTFA) containing 1% trimethylchlorosilane (TMCS) was added to the dry extracts followed by heating at 65°C for 20 minutes. One µL of each sample was injected into the gas chromatograph (GC) with a mass selective detector (MS).

Identification of Organic Residue using Gas Chromatography/Mass Spectrometry (GC/MS)
GC/MS analyses were carried out using a HP7890 gas chromatograph coupled to a HP5973 mass spectrometer (electron multiplier potential 2 KV, filament current 0.35 mA, electron energy 70 eV, and the spectra were recorded over the range m/z 40 to 800) using a split-less injection mode. An Agilent 7683 autosampler was used for sample introduction. Helium

was used as a carrier gas at a constant flow of 1.1 mL s–1. An isothermal hold at 50°C was kept for 2 minutes, followed by a heating gradient of 10°C min-1 to 320°C, with the final temperature held for 10 minutes. A 30 m, 0.25 mm ID 5% cross-linked phenylmethyl siloxane capillary column (HP-5MS) with a 0.25 μm film thickness was used for separation; the injection port temperature was 280°C and the MS interface temperature was 300°C. Peak assignments were carried out with the aid of library spectra (NIST 1.6) and compared with published data. The results were calibrated against known amounts of the internal standard (1-nonadecanol, C19Ol) added to each sample.

Results and Discussion

The complete molecular assemblage of the lipids extracted, their calibrated total amounts and their suggested interpretations are presented in Table 2.

Olive Oil

In all of the extracts of the items sampled, when no contamination was recognized, a clear and consistent predominance of palmitic acid (C16:0) over stearic acid (C18:0) is evident. This has been interpreted in the literature as an indication of plant oil (Copley et al. 2005; Baeten et al. 2013). Plant-derived sterols were also detected in some of the samples (see Table 2), indicating a vegetal component of the residues (Heron et al. 2010). Most plant oils cannot be further identified as many of them have similar fatty acid composition and ratios. One particular oil was identified as olive oil.

Olive oil has a fixed molecular assemblage consisting of free and bonded palmitic, linoleic, oleic and stearic acids, alongside β-sitosterol and glycerol (see Table 2). For pure olive oil, its free fatty acid composition consists of palmitic, stearic, oleic and linoleic acids, in relative abundances of C16:0> C18:0 and C18:1»C18:2>C18:0 formulas. After olive oil has been absorbed into ceramic vessels, its free fatty components relative abundance changes. Similar composition to that gained for the absorbed olive oil was detected in 18 of the 55 archaeological items analyzed. Our results support the accepted position that very high amounts of oleic acid (C18:1>55% of the total organic count) together with the relative priority of palmitic over stearic acid, accompanied with constant though low amounts of linoleic acid (C18:2) and the complete absence of linolenic (C18:3) acid, and the presence of β-sitosterol, the latter found in 3 out of the 18 vessels containing olive oil, all the presence of plant oil (Evershed et al. 1997)—specifically, olive oil (Boskou 2002; Koirala and Rosentreter 2009). Oleamide and oleonitrile, formed when olive oil is exposed to an alkali environment (Pecci and Cau-Ontiveros 2010), were also detected in a few extracts, along with monoolein (the triolein oxidation by-product). The normalized amount of linoleic acid may serve as a differential biomarker that can assist in distinguishing specific plant oils native to our region. Based on these variables, I conclude that olive oil was placed in numerous vessels found in the cave and that it was the most commonly used oil. *Olea europaea* is one of the most common tree species in the Mediterranean environment. Pollen

Table 2. Total Lipid Extracts of all the Studied Items from the Excavation[i]

Location	No.	Reg. No.	Type of Vessel	Lipid Analysis (by RT)	TOC (µg/g)	Identification
Dump	1	1061	Black piriform juglet	Thebaine, C7:0, ethylene glycol, C8:0, isoborneol, C9:0, C10:0, C11ol, vanillin, C12ol, C12:0, C13ol, vanillyl mandelic acid, n-C18, morphine, C14:0, n-C19, C15:0, n-C20, C16:0, n-C21, C18ol, n-C22, C18:2, C18:1, C18:0, n-C23, C20ol, n-C24, C20:0, WE, C22:0, MAG18:1, C24:0	40	Olive oil
	2	1075	Deep carinated bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	4	Olive oil
Group 1	3	1096	Dipper juglet	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	4	1093/2	Bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	15	Olive oil
	5	1000/5	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	Typical cont.
	6	1000/1	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	7	1091	Lamp bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	20	Olive oil
	8	1002/1	Jug	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	9	1002/2	Jug	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	10	1085	Black piriform juglet	C7:0, benzoic acid, C8:0, C9:0, benzaldehyde, C10:0, vanillin, C12:0, C13ol, vanillyl mandelic acid, C14:0, C16:1, C16:0, C18ol, C18:2, C18:1, C18:0, C19:0, C22ol, MAG18:1, C24:0	21	Olive oil
	11	1069	Jar	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	12	1067	Pithos	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
Group 2	13	1025	Black piriform juglet	C7:0, benzoic acid, C8:0, C9:0, benzaldehyde, C10:0, vanillin, C12:0, C13ol, vanillyl mandelic acid, C14:0, C16:1, C16:0, C18ol, C18:2, C18:1, C18:0, C19:0, C22ol, MAG18:1, C24:0	45	Olive oil
	14	1020/1	Twin vessel	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	15	1020/2	Twin vessel	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	16	1024	Piriform juglet	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	17	1098/1	Platter bowl	n/a		No lipids preserved
	18	1098/2	Platter bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, β-sitosterol	25	Olive oil

[i] Cx:y = a fatty acid with x carbons chain and y degree of unsaturation, all in their trimethylsilylated form; MAGx:y, TAGx:y = mono/tri-acylglycerol bounded with fatty acid with x carbons chain and y degree of unsaturation; Cxol = alcohol with x carbons chain; n-Cx = normal alkane with x carbons chain; cont. = contamination detected in extracts of vessels from the cave.

Table 2. (cont.)

Location	No.	Reg. No.	Type of Vessel	Lipid Analysis (by RT)	TOC (µg/g)	Identification
	19	1022	Platter closed bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	20	1023	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	21	1021	Bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	20	Olive oil
Group 3	22	1019	Deep carinated bowl	n/a		No lipids preserved
	23	1018	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	24	1017/1	Deep carinated bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	15	Olive oil
	25	1017/2	Deep carinated bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	15	Olive oil
Group 4	26	1040/1	juglet	C6:0, C8:0, C9:0, C10:0, benzaldehyde, C12:0, C16:0diacid, C14:0, C16:0, C18ol, C18:2, C18:1, C18:0	12	Plant oil
	27	1040/2	juglet	C6:0, C8:0, C9:0, benzaldehyde, C14:0, C16:1, C16:0, phytol, C18ol, C18:2, C18:1, C18:0	10	Plant oil
	28	1042	Deep carinated bowl	C6:0, C7:0, C8:0, glycerol, C9:0, benzaldehyde, C12:0, C14:0, C16:1, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, C22ol, MAG16:0, WE, C23:0	25	Plant oil
	29	1044	Deep carinated bowl	C9:0, C16ol, C16:1, C16:0, C18ol, C18:1, C18:0, dehydroabietic acid	20	Plant oil
	30	1030	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	18	cont.
	31	1036	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
Group 5	32	1008	Black piriform juglet	C6:0, C8:0, C9:0, benzaldehyde, C10:0, C12:0, C14:0, C16:0, C18:2, trans -C18:1, cis-C18:1, C18:0, isopimaric acid, tonalid, versalide, dehydroabietic acid, C21:0, C22ol, MAG16:0, C22:0, C24ol, C23:0, MAG18:1, MAG18:0, C24:0	29	cont. musk fragrance
	33	1007/1	Dipper juglet	n/a		No lipids preserved
	34	1007/2	Bowl	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	20	Olive oil
	35	1006	Jar	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	20	Olive oil

Table 2. (cont.)

Location	No.	Reg. No.	Type of Vessel	Lipid Analysis (by RT)	TOC (µg/g)	Identification
Group 6	36	1090	Piriform juglet	C6:0, C7:0, C8:0, C9:0, benzaldehyde, C14:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0	30	Olive oil
	37	1083	Dipper juglet	C6:0, C8:0, C9:0, C16:0, C18:2, trans-C18:1, cis-C18:1, C18:0, C20:0, C22ol, C22:0, C24ol, C24:0	50	Olive oil
	38	1088/1	Piriform juglet	C6:0, C7:0, C8:0, glycerol, C9:0, benzaldehyde, C12:0, C13:0, C14:0, C16:0, C18:2, C18:1, C18:0, MAG18:2, C20:0, MAG16:0, C22:0, MAG18:1	20	Olive oil + other plant essence
	39	1088/2	Piriform juglet	C6:0, C8:0, C9:0, C14:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, MAG16:0, C22:0, MAG18:1	30	Olive oil
	40	1072	Platter bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	41	1081	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	42	1073	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	43	1076	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	44	1089	Deep carinated bowl	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	45	1082	Deep carinated bowl	n/a		No lipids preserved
	46	1066	Pithos	C8:0, glycerol, C9:0, C16:0, C18ol, C18:2, C18:1, C18:0, C20:0, β-sitosterol	10	Olive oil
	47	1068	Pithos	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.
	48	1064	Bowl on a stand	C6:0, C8:0, C9:0, C14:0, C16:1, C16:0, C18ol, C18:2, C18:1, C18:0	20	Olive oil
L99	49	1077	Cooking pot	C16:1, C16:0, C18ol, C18:1, C18:0	>10	cont.

histograms show very clearly that olive trees were most prevalent during the Chalcolithic period (fifth millennium BCE; Langgut, Adams and Finkelstein 2016).

Vanillin

Three black juglets were sampled in different areas along their profile (one was sampled close to its rim and the other two, close to their base due to restoration considerations). Their extraction results are very similar (Tables 1; 2:1, 10, 13). Apparently, all three small piriform black juglets from this MB II cave contained vanillin mixed with olive oil. The source of the vanillin is unclear. Although intuitively its source would be vanilla pods, in which vanillin is highly abundant (Joel et al. 2003; Brillouet et al. 2014; Linares et al. 2019), this may not be the case.

Little is known about the origin of vanilla cultivation in antiquity, before it was cultivated a mere 300 years ago by the Pre-Columbian Mesoamerican Aztecs (Lubinsky et al. 2008; Teoh 2019). Vanilla is a tropical climbing vine which grows best in a hot and humid climate, at a height ranging from 0–1500 m, idealy with a moderate rainfall of 1500–3000 mm, evenly distributed throughout 10 months of the year. Optimum temperatures for cultivation are 15–30°C during the day and 15–20°C at night. Ideal humidity is around 80% (Korthou and Verpoorte 2007; Chambers 2018). These conditions hint to its place of origin, perhaps in regions near the Indian Ocean, including Madagascar (Roux-Cuvelier and Grisoni 2010).

The various subspecies of vanilla known today are *Vanilla planifolia* (syn. *V. fragrans*), grown on Madagascar, Réunion, and other tropical areas along the Indian Oceant; *V. tahitensis*, grown in the South Pacific; and *V. pompona*, found in the West Indies and Central and South America (Fouché and Jouve 1999; Besse et al. 2004). The majority of the world's vanilla is the *V. planifolia* species, more commonly known as Bourbon vanilla (after the former name of Réunion, Île Bourbon) or Madagascar vanilla, which is produced in Madagascar and neighboring islands in the southwestern Indian Ocean, and in Indonesia (Teoh 2019).

It seems that initial attempts to cultivate vanilla outside Mexico and Central America proved futile because of the symbiotic relationship between the vanilla orchid and its natural pollinator, the local species of *Melipona* bee (Lubinsky, Van Dam and Van Dam 2009).[1] This bee-flower symbiosis provided Mexico with a 300-year-long monopoly on vanilla production, from the time it was first discovered by Europeans. Moreover, the chemical composition of vanilla, found in the seeds of the plant, is an extremely complicated mixture of 171 identified aromatic components (Burdock 2005). Therefore, suggesting that the vanillin found in the juglets derives from vanilla pods is not sound.

Another explanation, based on the synthetic production from a more readily available natural compound such as eugenol, guaiacol or lignin (Hazen 1995; Gassenheimer and Binggeli 2008), is that the source of the vanillin in the juglets derives from modern contamination. This possibility is equally difficult to accept, as vanillin was detected in only three of a large assemblage of 55 items. These three juglets were found in three different loci in the cave, suggesting that the vanillin detected in those juglets is genuine.

I suggest that the source for the vanillin extract in the juglets is the result of enzymatic or bacterial activity, enabled in the climatic conditions that prevailed within the cave. There are a variety of microorganisms that are known to naturally produce vanillin in very large amounts. These microorganisms, in a warm (35°C), humid and alkaline (pH 9.0)

[1] Pollination is required as *V. Planifolia* flowers are hermaphroditic: they carry both male (anther) and female (stigma) organs; however, to avoid self-pollination, a membrane separates those organs. The flowers can be naturally pollinated only by bees of the Melipona genus found in Mexico.

environment, can readily convert many natural compounds such as ferulic acid,[2] vanillic acid, eugenol, isoeugenol, phenolic stilbenes, aromatic amino acids, etc., into vanillin (Heuvel et al. 2001; Vaithanomsat and Apiwatanapiwat 2009; Zamzuri and Abd-Aziz 2013; Chen et al. 2016). These precursors are abundant in many agricultural crops, such as rice, wheat, maize and sugar beet (Karmakar et al. 2000; Di Gioia et al. 2011).

Vanillin production via the conversion of ferulic acid, for example, has been widely reported in various microorganisms. Thus, the warm and moist conditions of the burial cave, together with the alkaline environment produced in it (as demonstrated by the formation of oleanitrile and oleamide from the olive oil [Pecci and Cau-Ontiveros 2010]), would enable the spontaneous and natural production of vanillin by any of the microorganisms listed here or others (Chen et al. 2016). Therefore, I suggest that the vanillin present in the juglets is the by-product of such microbial activity.

Musk Contamination

The molecular composition of the extract from the content of the juglet in L1008 (see Tables 1; 2:32) may indicate that it was musk oil. Although Egyptian musk oil made from local plants contained musky-flavored oil and was known in antiquity, all musk-related compounds (mainly tonalid and versalide) detected in this juglet are synthetic and are widely used in the cosmetics industry (Kraft 2004; Sommer 2004). Isopimaric acid, also detected in the extract, originates from many trees, especially conifers, which are abundant in the environment of the cave. These contaminating compounds could have originated from any modern source that was in contact with the sample during the excavation.

CONCLUSIONS

In 20 of the 49 vessels sampled, it was possible to recognize the presence of plant oil, namely olive oil. No other organic substance was detected. The results of this examination and analysis indicate that the residues were the result of contamination, a frequent occurrence in burial caves, where olive oil and local environmental conditions can serve as a fruitful catalyzer for the development of large quantities of vanillin and other related compounds.

[2] These include *Pseudomonas acidovorans, Saccharomyces cerevisiae, Rhodotorula rubra, Streptomyces setonii, Bacillus coagulans, Streptomyces halstedii, Schizophyllum commune, Bacillus licheniformis, Delftia acidovorans, Pseudomonas putida* and *Sphingomonas paucimobilis* (Yoon et al. 2005; Ghosh et al. 2007; Zheng et al. 2007; Abdelkafi et al. 2008; Tsujiyama and Ueno 2008; Cortez and Roberto 2010; Ji et al. 2011).

References

Abdelkafi S., Labat M., Gam Z.B.A., Lorquin J., Casalot L. and Sayadi S. 2008. Optimized Conditions for the Synthesis of Vanillic Acid under Hypersaline Conditions by *Halomonas elongata* DSM 2581T Resting Cells. *World Journal of Microbiology and Biotechnology* 24:675–680.

Baeten J., Jervis B., De Vos D. and Waelkens M. 2013. Molecular Evidence for the Mixing of Meat, Fish and Vegetables in Anglo-Saxon Coarseware from Hamwic, UK. *Archaeometry* 55:1150–1174.

Ben-Ari N. and Wiegmann A. This volume. A Middle Bronze Age II Burial Cave at Beit Ṣafafa, Jerusalem.

Besse P., Silva D., Bory S., Grisoni M., Le Bellec F. and Duval M.F. 2004. RAPD Genetic Diversity in Cultivated Vanilla: Vanilla Planifolia, and Relationships with V. Tahitensis and V. Pompona. *Plant Science* 167:379–385.

Boskou F.D. 2002. Olive Oil. In F. Gunstone ed. *Vegetable Oils in Food Technology: Composition, Properties and Uses*. Oxford. Pp. 244–277.

Brillouet J.-M., Verdeil J.-L., Odoux E., Lartaud M., Grisoni M. and Conéjéro G. 2014. Phenol Homeostasis is Ensured in Vanilla Fruit by Storage under Solid Form in a New Chloroplast-Derived Organelle, the Phenyloplast. *Journal of Experimental Botany* 65:2427–2435.

Burdock G.A. 2005. *Fenaroli's Handbook of Flavor Ingredients*. Boca Raton.

Chambers A.H. 2018. *Establishing Vanilla Production and a Vanilla Breeding Program in the Southern United States.* In D. Havkin-Frenkel and F.C. Belanger eds. *Handbook of Vanilla Science and Technology*. Hoboken, N.J. Pp. 165–180.

Chen P., Yan L., Wu Z., Li S., Bai Z., Yan X., Wang N., Liang N. and Li H. et al. 2016. A Microbial Transformation using Bacillus subtilis B7-S to Produce Natural Vanillin from Ferulic Acid. *Scientific Reports* 6:20400.

Copley M.S., Bland H.A., Rose P., Horton M. and Evershed R.P. 2005. Gas Chromatographic, Mass Spectrometric and Stable Carbon Isotopic Investigations of Organic Residues of Plant Oils and Animal Fats Employed as Illuminants in Archaeological Lamps from Egypt. *Analyst* 130:860–871.

Cortez D.V. and Roberto I.C. 2010. Individual and Interaction Effects of Vanillin and Syringaldehyde on the Xylitol Formation by *Candida guilliermondii*. *Bioresource Technology* 101:1858–1865.

Di Gioia D., Luziatelli F., Negroni A., Ficca A.G., Fava F. and Ruzzi M. 2011. Metabolic Engineering of *Pseudomonas fluorescens* for the Production of Vanillin from Ferulic Acid. *Journal of Biotechnology* 156:309–316.

Evershed R.P., Vaughan S. J., Dudd S.N. and Soles J.S. 1997. Fuel for Thought? Beeswax in Lamps and Conical Cups from Late Minoan Crete. *Antiquity* 71:979–985.

Fouché J.G. and Jouve L. 1999. Vanilla Planifolia: History, Botany and Culture in Reunion Island. *Agronomie* 19/8:689–703.

Gassenheimer K. and Binggeli E. 2008. Vanilla Bean Quality: A Flavour Industry View. In I. Blank, M. Wüst and C. Yeretzian eds. *Expression of Multidisciplinary Flavour Science (Proceedings of the 12th Weurman Symposium, Interlaken, Switzerland 2008).* Wädenswil. Pp. 203–206.

Ghosh S., Sachan A., Sen S.K. and Mitra A. 2007. Microbial Transformation of Ferulic Acid to Vanillic Acid by *Streptomyces sannanensis* MTCC 6637. *Journal of Industrial Microbiology and Biotechnology* 34:131–138.

Hazen J. 1995. *Vanilla*. San Francisco.

Heron C., Nilsen G., Stern B., Craig O. and Nordby C. 2010. Application of Lipid Biomarker Analysis to Evaluate the Function of 'Slab-Lined Pits' in Arctic Norway. *JAS* 37:2188–2197.

Heuvel R.H. van den, Fraaije M.W., Laane C. and Berkel W.J. van. 2001. Enzymatic Synthesis of Vanillin. *Journal of Agricultural Food Chemistry* 49:2954–2958.

Ji L., Shen Y., Xu L., Peng B., Xiao Y. and Bao X. 2011. Enhanced resistance of *Saccharomyces cerevisiae* to Vanillin by Expression of *lacA* from *Trametes* sp. AH28-2. *Bioresource Technology* 102:8105–8109.

Joel D.M., French J.C., Graft N., Kourteva G., Dixon R.A., and Havkin-Frenkel D. 2003. A Hairy Tissue Produces Vanillin. *Israel Journal of Plant Science* 51:157–159.

Karmakar B., Vohra R.M., Nandanwar H., Sharma P., Gupta K.G. and Sobti R.C. 2000. Rapid Degradation of Ferulic Acid via 4-vinylguaiacol and Vanillin by a Newly Isolated Strain of *Bacillus coagulans*. *Journal of Biotechnology* 80:195–202.

Koirala B. and Rosentreter J. 2009. Examination of Prehistoric Artifacts via Fatty Acid Methyl Ester (FAME) Techniques using Modern Environmental Stewardship. *JAS* 36:1229–1242.

Korthou H. and Verpoorte R. 2007. Vanilla. In R.G. Berger ed. *Flavours and Fragrances: Chemistry, Bioprocessing and Sustainability*. Berlin–Heidelberg. Pp. 203–217.

Kraft P. 2004. Aroma Chemicals IV: Musks. In D.J. Rowe ed. *Chemistry and Technology of Flavours and Fragrances*. New York. Pp. 143–168.

Langgut, D., Adams M. J. and Finkelstein I. 2016. Climate, Settlement Patterns and Olive Horticulture in the Southern Levant during the Early Bronze and Intermediate Bronze Ages (c. 3600–1950 BC). *Levant* 48:117–134.

Linares V., Adams M.J., Cradic M.S., Finkelstein I., Lipschits O., Martin M.A.S., Neumann R., Stockhammer P.W. and Gadot Y. 2019. First Evidence for Vanillin in the Old World: Its Use as Mortuary Offering in Middle Bronze Canaan. *JAS Reports* 25:77–84.

Lubinsky P., Bory S., Hernández J.H., Kim S.-C. and Gómez-Pompa A. 2008. Origins and Dispersal of Cultivated Vanilla (*Vanilla planifolia* Jacks.[Orchidaceae]). *Economic Botany* 62:127–138.

Lubinsky P., Van Dam M. and Van Dam A. 2009. Pollination of Vanilla and Evolution in Orchidaceae. *Lindleyana* 75:926–929.

Pecci A. and Cau-Ontiveros M.A. 2010. Análisis de residuos orgánicos en ánforas: El problema de la resina y el aceite. In J.M. Blázquez and J. Remesal eds. *Estudios sobre el Monte Testaccio (Roma)* V (Instrumenta 35). Barcelona. Pp. 593–600.

Roux-Cuvelier M. and Grisoni M. 2010. Conservation and Movement of Vanilla Germplasm. In E. Odoux and M. Grisoni eds. *Vanilla*. Boca Raton. Pp. 31–41

Sommer C. 2004. The Role of Musk and Musk Compounds in the Fragrance Industry. In G.G. Rimkus ed. *Synthetic Musk Fragrances in the Environment*. Berlin. Pp. 1–16.

Teoh E.S. 2019. The Story of Vanilla. In E.S. Teoh ed. *Orchids as Aphrodisiac, Medicine or Food*. Cham. Pp. 109–130.

Tsujiyama S. and Ueno M. 2008. Formation of 4-Vinyl Guaiacol as an Intermediate in Bioconversion of Ferulic Acid by *Schizophyllum commune. Bioscience, Biotechnology and Biochemistry* 72:212–215.

Vaithanomsat P. and Apiwatanapiwat W. 2009. Feasibility Study on Vanillin Production from *Jatropha curcas* Stem using Steam Explosion as a Pretreatment. *International Journal of Agricultural and Biosystems Engineering* 3:258–261.

Yoon S.-H., Li C., Lee Y.M., Lee S.H., Kim S.H., Choi M.S., Seo W.-T., Yang J.-K., Kim J.-Y. and Kim S.W. 2005. Production of Vanillin from Ferulic Acid using Recombinant Strains of *Escherichia coli. Biotechnology and Bioprocess Engineering* 10:378–384.

Zamzuri N.A. and Abd-Aziz S. 2013. Biovanillin from Agro Wastes as an Alternative Food Flavour. *Journal of Science of Food and Agriculture* 93:429–438.

Zheng L., Zheng P., Sun Z., Bai Y., Wang J. and Guo X. 2007. Production of Vanillin from Waste Residue of Rice Bran Oil by *Aspergillus niger* and *Pycnoporus cinnabarinus. Bioresource Technology* 98:1115–1119.