CHEMICAL EVIDENCE FOR A STRUCTURED AGRICULTURAL MANURING REGIME ON THE ISLAND OF PSEIRA, CRETE DURING THE MINOAN PERIOD

Introduction

The Island of Pseira is located off the north-eastern coast of Crete. Measuring about two kilometres in length, it has a terrain composed predominantly of steep hills and sheer, precipitous cliffs. Initial human habitation of the island, on the south-eastern coast, is dated to the Final Neolithic with a greatly expanded populous by the Late Minoan period. At this time catastrophic events led to the destruction of the settlement on the island which remained mostly uninhabited until the early Byzantine period.[1] Formations of soil on Pseira are extremely dry, calcareous and shallow. However, the use of fertile land in this marginal environment was maximized through the construction of terraces. Historical agricultural activity on Pseira Island may be divided into three time periods. Over the first period, from the Final Neolithic to the Middle Bronze Age, there was a gradual increase in the use of land available both naturally and artificially, reclaimed through the process of terracing. In the second period, dating to Late Minoan I, land-use increased still further as a result of settlement expansion, and two stone dams were built across ravines. Finally, after a period of abandonment, a bout of agricultural cultivation occurred in Late Minoan III; however, there is no evidence regarding a resumption of the intensive practices previously used. The presence of potsherds and other occupation debris has been noted in soils associated with the first and second periods of agricultural land-use. In a study concerning similar distributional scatters of potsherds, it has been proposed that they originated from a systematic collection of animal and human excrement, together with household rubbish, followed by a regular spread of the mixture across the cultivated landscape as fertiliser.[2] This would explain the potsherd scatter observed in the terrace soils as most likely the result of village wastes and water being regularly transported to the terraces. The building of dams in the second time period would have facilitated the agricultural use of water as well as provided a probable terrace input in the form of silt collected from behind the two dams. Tillage in the first time period was particularly deep with Early to Middle Minoan sherds existing in soils just above bedrock. During the third period of agricultural use the absence of associated potsherd scatter infers that this process of fertilisation was most likely discontinued, although the population associated with this time period would also have been much smaller.[3]

To date a number of methods have been utilised to detect ancient manuring practices. These include the use of phosphorous concentration,[4] micromorphology,[5] potsherd scatter,[6] and magnetic susceptibility.[7] More recently an alternative approach to identifying the

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1 P.P. BETANCOURT and R. HOPE SIMPSON, " The Agricultural System of Bronze Age Pseira," *Cretan Studies* 3 (1992) 47-54.

2 J.L. BINTLIFF and A.M. SNODGRASS, "Off-site pottery distributions: a regional and interregional perspective," *Current Anthropology* 29 (1988) 506-513.

3 BETANCOURT and HOPE SIMPSON (*supra* n. 1).

4 R.C. EIDT, "Advances in Abandoned Settlement Analysis: Application to Prehistoric Anthrosols in Columbia, South America," Ph.D. Dissertation (1984) University of Winsconsin, Milwaukee; L. PROSCH-DANIELSEN and A. SIMONSEN, "Principal components analysis of pollen, charcoal and soil phosphate data as a tool in prehistoric land-use investigation at Forsandmoen, SW Norway," *NorwegArchaeolRev*. 21 (1988) 85-102.

5 S. LIMBREY, *Soil Science and Archaeology* (1975).

6 BINTLIFF and SNODGRASS (*supra* n. 2); M.A. COURTY, P. GOLDBERG and R. MACPHAIL, *Soils and micromorphology in archaeology* (1989).

7 G.E. MULLEN, "Magnetic susceptibility of the soil and its significance in soil science – a review," *JSoilSci* 28 (1977) 223-246.

systematic deposition of faecal matter, involving the detection of 5β-stanols, has proven to be particularly successful.[8] Pl. XIa shows how deposition of faecal material can produce an enhancement of 5β-stanols relative to the background of 5β-stanols in a control soil. Analyses for these compounds have also been used to determine faecal inputs to archaeological soil deposits.[9] Furthermore, the technique has been extended so that information regarding the faecal source may be determined by considering the relative abundances of 5β-stanol homologues.[10] The use of these highly diagnostic decay resistant biomarkers offers a direct and robust means of detecting faecal manure inputs to archaeological soils.

5β-Cholestan-3β-ol (coprostanol; **2**) is a sterol reduction product of cholest-5-en-3β-ol (cholesterol; **1**), its most common origin being a microbially mediated hydrogenation in the intestinal tracts of most higher mammals.[11] The intimate association of coprostanol with faeces has also made it a useful analytical tracer in a wide range of additional applications including medical and modern-day pollution studies.[12] Using coprostanol and several related stanols, we provide here chemical evidence that a structured agricultural manuring regime once operated in ancient Crete from a time as early as ca. 2500 B.C.

Sample Site

Samples were collected from the profile of a trench dug in soil retained behind the wall of an ancient agricultural terrace (Terrace G2) located on Pseira island in the predominantly limestone area north of the main archaeological site (onetime south-eastern settlement). The terrace was on a 25° slope, 90 m above sea level; overlying vegetation was scarce and the soil very dry when sampled. Samples were taken at depths of 0, 15, 25, 35, 40, 50, 60, 70, 80, 85 and 95 cm and a control sample was obtained from the site of an archaeological building; this was as unlikely to contain manure as any other control sample which could be collected on the island.

Consideration of total organic carbon (TOC) levels

Pl. XIb depicts the results of analyses performed to ascertain levels of total organic carbon in the profile of Terrace G2. As can be seen, the quantity of organic carbon is highest

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8 R.P. EVERSHED and P.H. BETHELL, "Application of multimolecular biomarker techniques to the identification of faecal material in archaeological soils and sediments," *ACS Symposium Series* 625 (1996) 157-172; R.P. EVERSHED, P.H. BETHELL, P. REYNOLDS and L.J. GOAD, "5β-Stigmastanol and related 5β-stanols as biomarkers of manuring: Analysis of modern experimental material and assessment of the archaeological potential.," *JArchSci* 24 (1997) 485-495; I.D. BULL. P.F. van BERGEN, P.R. POULTON and R.P. EVERSHED, "Organic geochemical studies of soils from the Rothamsted Classical Experiments II: Soils from the Hoosfield Spring Barley experiment treated with different quantities of manure," *Org Geochem.*, in press.

9 B.A. KNIGHTS, C.A. DICKSON, B.H. DICKSON and D.H. BREEZE, "Evidence concerning the Roman military diet at Bearsden, Scotland, in the 2nd century AD," *JArchSci* 10 (1983) 139-152; C. PEPE, P. DIZABO, P. SCIBE, J. DAGAUX and A. SALIOT, "Les marqueurs biogéochimiques: application à l'archéologie," *Revue d'Archéométrie* 13 (1989) 1-11; C. PEPE and P. DIZABO, "Étude d'une fosse du XIIIe siècle par les marqueurs biogéochimiques: chantier archéologique du Louvre (Paris)," *Revue d'Archéométrie* 14 (1990) 23-28.

10 P.H. BETHELL, L.J. GOAD, R.P. EVERSHED and J. OTTAWAY, "The study of molecular markers of human activity: The use of coprostanol in the soil as an indicator of human faecal material.," *JArchSci* 21 (1994) 619-632; I.A. SIMPSON, S.J. DOCKRILL, I.D. BULL and R.P. EVERSHED, "Early anthropogenic soil formation at Tofts Ness, Sanday, Orkney," *JArchSci*, in press.

11 J.J. MURTAUGH and R.L. BUNCH, "Sterols as a measure of fecal pollution," *JWatPollutControl* 39 (1967) 404-409; P.G. HATCHER and P.A. MCGILLIVRAY, "Sewage contamination in the New York Bight. Coprostanol as an indicator," *EnvirSciTechnol* 13 (1979) 1225-1229.

12 G.M. BARKER, S. RADLEY, A. DAVIS, K.D.R. SETCHELL, N. O'CONNELL, I.A. DONOVAN, M.R.B. KEIGHLEY and J.P. NEOPTOLEMOS, "Analysis of faecal neutral sterols in patients with familial adenomatous polyposis by gas chromatography-mass spectrometry," *IntJColorectalDisease* 8 (1993) 188-192; J.O. GRIMALT, P. FERNANDEZ, J.M. BAYONA and J. ALBAIGES, "Assessment of faecal sterols and ketones as indicators of urban sewage inputs to coastal waters," *EnvirSciTechnol* 24/3 (1990) 357-363.

in the sample from 15 cm depth (2.3 %), rising from a surficial level of 1.9 %. Samples from >15 cm depth exhibit a rapid decrease in TOC with all values at >35 cm depth residing in the 0.6-0.9 % range. The TOC content of the profile is relatively low compared with an average TOC of 5 % for mineral soils.[13] The higher values obtained from the shallow samples can be attributed to input of organic matter from modern-day vegetation, with the highest value at 15 cm depth resulting from the intimate association of rhizomaceous matter. These results provide little evidence of recent disturbance of the archaeological soil since major bioturbation would have resulted in higher TOC inclusion at lower depths. TOC measurements are, however, unable to provide information regarding the source and/or age of specific inputs.

Analysis of the soil sterols

Sterol components were isolated from soils by a combination of solvent extraction, chromatographic and chemical inclusion techniques. Characterisation of sterols was made using gas chromatography/mass spectrometry (GC/MS). The major sterol components observed in each soil are cholesterol and 24-ethylcholest-5-en-3β-ol (sitosterol; **5**); however, the sterol fraction at 85 cm depth contains coprostanol as a major component, significantly more abundant than the usual diagenetic reduction product of cholesterol, 5α-cholestan-3β-ol (5α-cholestanol; **4**), infering possible manure deposition. Furthermore another 5β-stanol, 24-ethyl-5β-cholestan-3β-ol (5β-stigmastanol; **6**) resides at much lower abundance than would be expected for a ruminant faecal source indicating that relatively low quantities of the plant derived sterol, sitosterol, were ingested by the putative mammalian source thereby inferring a human or porcine origin for the manure.[14]

Modern-day studies of sewage pollution have shown a stanol ratio [**2**/(**2**+**4**)] to be a reliable proxy for concentration in the detection of faecal deposition; a theoretical ratio of >0.7 has been ascribed as a lower limit for definite faecal deposition.[15] Using this ratio circumvents the problem of a natural 5β-stanol background in soils since the criterion for faecal deposition no longer involves the absolute concentration of these compounds. Moreover, we propose that this parameter may be further improved by including 5β-cholestan-3α-ol (epicoprostanol; **3**) which under certain conditions, e.g. anaerobic sewage sludge digestion, may be produced by a microbially mediated epimerisation of coprostanol.[16] Application of this modified ratio [(**2**+**3**)/(**2**+**3**+**4**)] to sterol data obtained from the terrace soils reveals the periodic occurrence of manuring episodes (>0.7) at sub-surficial depths (Pl. XIIa). These chemical data correlate with the occurrence of pottery sherds (potsherds), considered to be an archaeological artefact of intensive manuring regimes, throughout the majority of the profile (>20 cm depth).[17] Potsherds at 20-45 cm have been placed chronologically in the Late Minoan (1700-1400 B.C.) and earlier whilst sub-45 cm sherds date from Early (2500-2000 B.C.) to Middle Minoan (2000-1700 B.C.).[18] Inspection of the ratio of coprostanol to epicoprostanol in the same samples reveals high values (> 2) in the deepest soils (85 and 95 cm) and is indicative of minimal post-excretive reworking of coprostanol, and hence, the faecal material (Pl. XIIb).[19] In contrast, the relatively high amounts of epicoprostanol in shallower soils (e.g. 35 and 40 cm) infers possible pre-depositional conditions which have resulted in the further epimerisation of coprostanol.

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13 E.M. BRIDGES, *World Soils* (2nd ed., 1978).
14 BETHELL, GOAD, EVERSHED and OTTAWAY (*supra* n. 10).
15 GRIMALT, FERNANDEZ, BAYONA and ALBAIGES (*supra* n. 12).
16 D.V. MCCALLEY, M. COOKE and G. NICKLESS, "Effect of sewage treatment on faecal sterols," *WaterRes* 15 (1981) 1019-1025; A.S. MACKENZIE, S.C. BRASSELL, G. EGLINGTON and J.R. MAXWELL, "Chemical fossils: The geological fate of steroids," *Science* 217 (1982) 491-504.
17 BINTLIFF and SNODGRASS (*supra* n. 2).
18 BETANCOURT and HOPE SIMPSON (*supra* n. 1).
19 I.D. BULL, "New Molecular Methods for Tracing Natural and Anthropogenic Inputs to Soils and Sediments," Ph.D Dissertation (1997) University of Bristol.

Archaeological implications

In light of these results it can be seen that an organised manuring regime of agricultural terrace soils was practised on Pseira Island from the Early to Late Minoan periods. Manure would have been formed from excrement (probably human given that there exists no evidence of pig husbandry) and household waste. The manure was most likely widely used and in short supply during the earlier time period corresponding with the deepest soils. In later periods the increase in population would have led to greater waste production and hence a greater possibility of significant accretion of manure, as midden piles, before transportation to the terraces. Such piles may well have been a very favorable environment for the production of epicoprostanol. As far as we are aware, this is the oldest (ca. 4500 years) structured manuring regime yet detected via the analysis of coprostanol and related compounds. Moreover, we also believe this to be the first positive correlation between faecal sterol analysis and the occurrence of potsherd scatter thereby strongly supporting both techniques as analytical tools to be used in the detection of archaeological deposition of faecal material.

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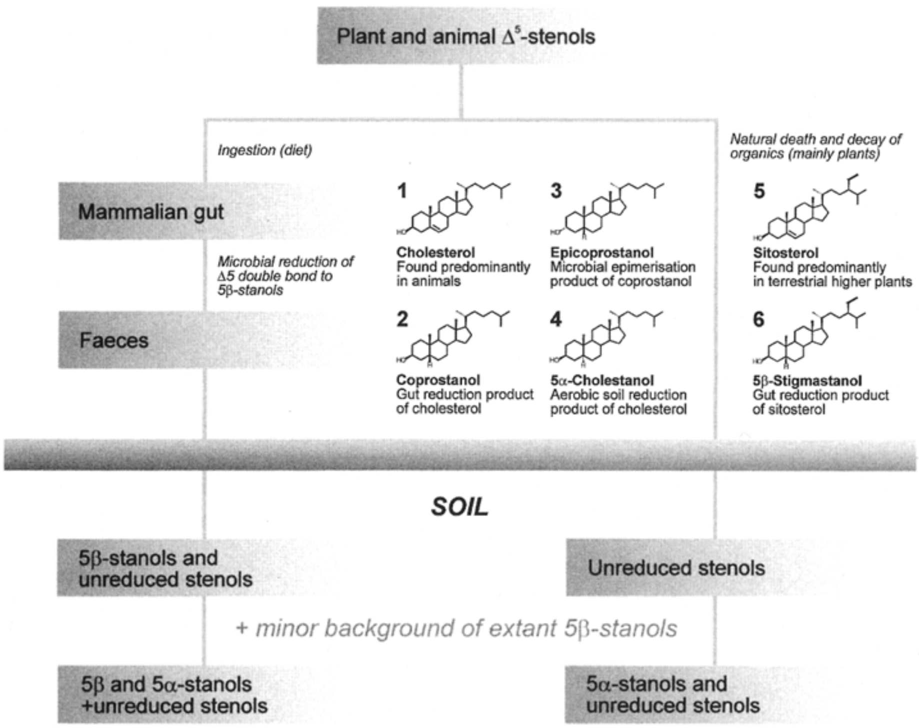
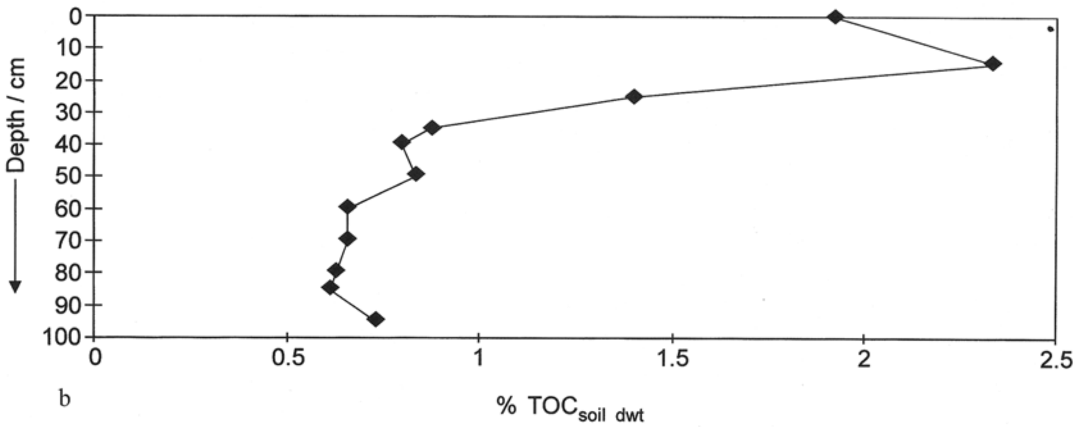
LIST OF ILLUSTRATIONS

XI



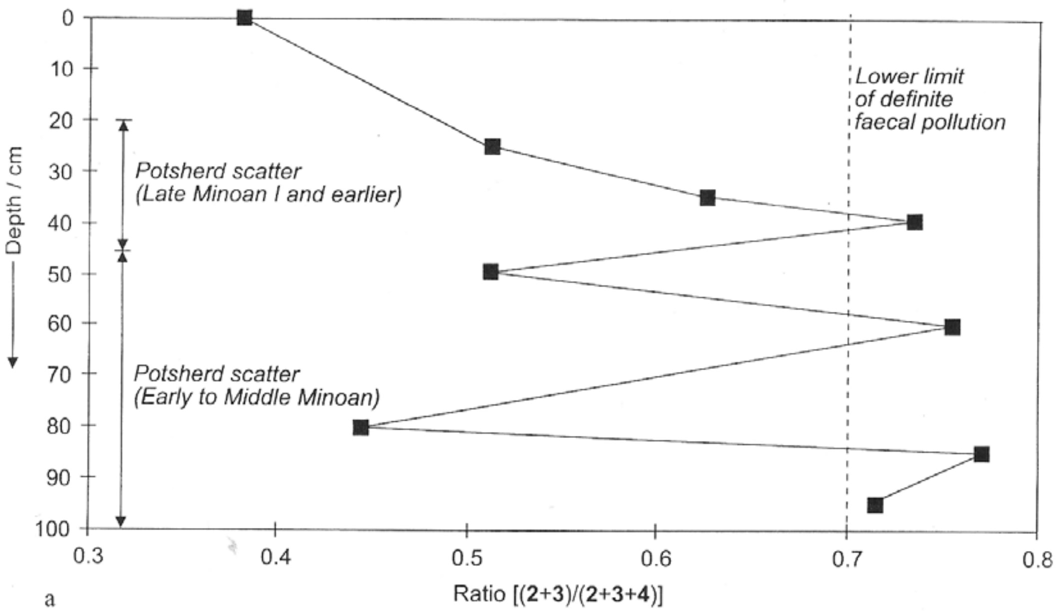
Plant and animal Δ5-stenols
Ingestion (diet)
Natural death and decay of organics (mainly plants)
Mammalian gut
Microbial reduction of Δ5 double bond to 5β-stanols
Faeces
1
Cholesterol
Found predominantly in animals
3
Epicoprostanol
Microbial epimerisation product of coprostanol
5
Sitosterol
Found predominantly in terrestrial higher plants
2
Coprostanol
Gut reduction product of cholesterol
4
5α-Cholestanol
Aerobic soil reduction product of cholesterol
6
5β-Stigmastanol
Gut reduction product of sitosterol
SOIL
5β-stanols and unreduced stenols
Unreduced stenols
+ minor background of extant 5β-stanols
5β and 5α-stanols +unreduced stenols
5α-stanols and unreduced stenols

a



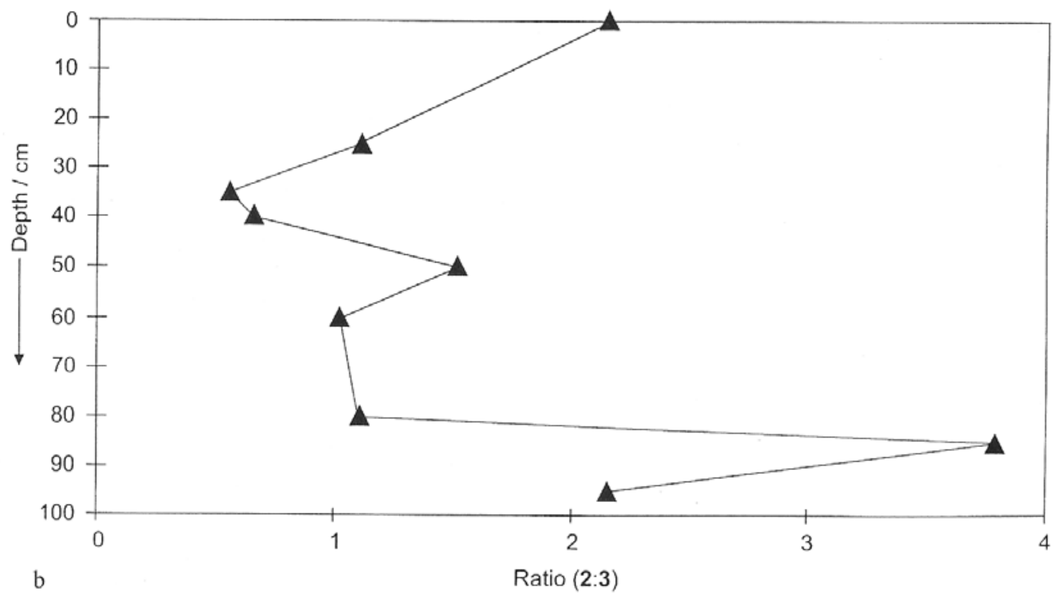
Depth / cm
0
10
20
30
40
50
60
70
80
90
100
0
0.5
1
1.5
2
2.5
% TOC soil dwt

b



Depth / cm
0
10
20
30
40
50
60
70
80
90
100
Potsherd scatter
(Late Minoan I and earlier)
Potsherd scatter
(Early to Middle Minoan)
Lower limit
of definite
faecal pollution
0.3
0.4
0.5
0.6
0.7
0.8
Ratio [(2+3)/(2+3+4)]

a



Depth / cm
0
10
20
30
40
50
60
70
80
90
100
0
1
2
3
4
Ratio (2:3)

b