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► **To cite this version:**

Renata Zocatelli, Jean-Robert Disnar, Jérémy Jacob, Claude Le Milbeau. STEROLS AND BILE ACIDS IN URBAN AND RURAL SOILS AS FAECAL MARKERS OF LAND-USE SINCE THE BRONZE AGE. IMOG2013: the 26th International Meeting on Organic Geochemistry, Sep 2013, Tenerife, Spain. insu-00866040

HAL Id: insu-00866040

<https://insu.hal.science/insu-00866040v1>

Submitted on 31 Mar 2016

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STEROLS AND BILE ACIDS IN URBAN AND RURAL SOILS AS FAECAL MARKERS OF LAND-USE SINCE THE BRONZE AGE

Renata Zocatelli, Jean-Robert Disnar, Jérémy Jacob, Claude Le Milbeau

Université d'Orléans, Institut des Sciences de la Terre d'Orléans (ISTO), CNRS/INSU, BRGM - UMR 7327, F45071 Orléans Cedex 2, France.

Section A18
Poster 508

Introduction and aims

Faecal biomarkers (C27, C28 and C29 coprostanols and bile acids) found in soils and sediments have been proposed as direct indicators of human presence or of breeding since these compounds are formed in the intestinal tracts of higher mammals (Elhmmali et al, 2000, Bull et al., 2002).

The goal of this study is to test the potentialities of faecal biomarkers in soils and sediments to resolve, at the field-scale, the strategies of land use. For this purpose, we designed our study in three steps:

- i) characterization of faecal biomarkers imprints of source animals,
- ii) evaluation of the faecal biomarkers stability through the analysis of samples of different age,
- iii) examination of the distribution of faecal biomarkers in a wide diversity of contexts under different type of soils, different land-uses and degrees of anthropization.

Methodology

Dried and crushed samples (~5 g) were extracted using accelerated solvent extraction with $\text{CH}_2\text{Cl}_2:\text{MeOH}$ (1:1 v/v; ASE 200 Dionex®). Total extract was fractionated into neutral and acidic compounds using solid phase extraction on Aminopropyl Bond Elute® phase according to Jacob et al. (2005). Acid fractions were methylated (anhydrous MeOH and acetyl chloride). Then, sterols and acidic fractions were further derivatized by BSTFA. Standards (5 α -cholestane and nor-deoxycholic acid) were added prior to GC-MS analysis.

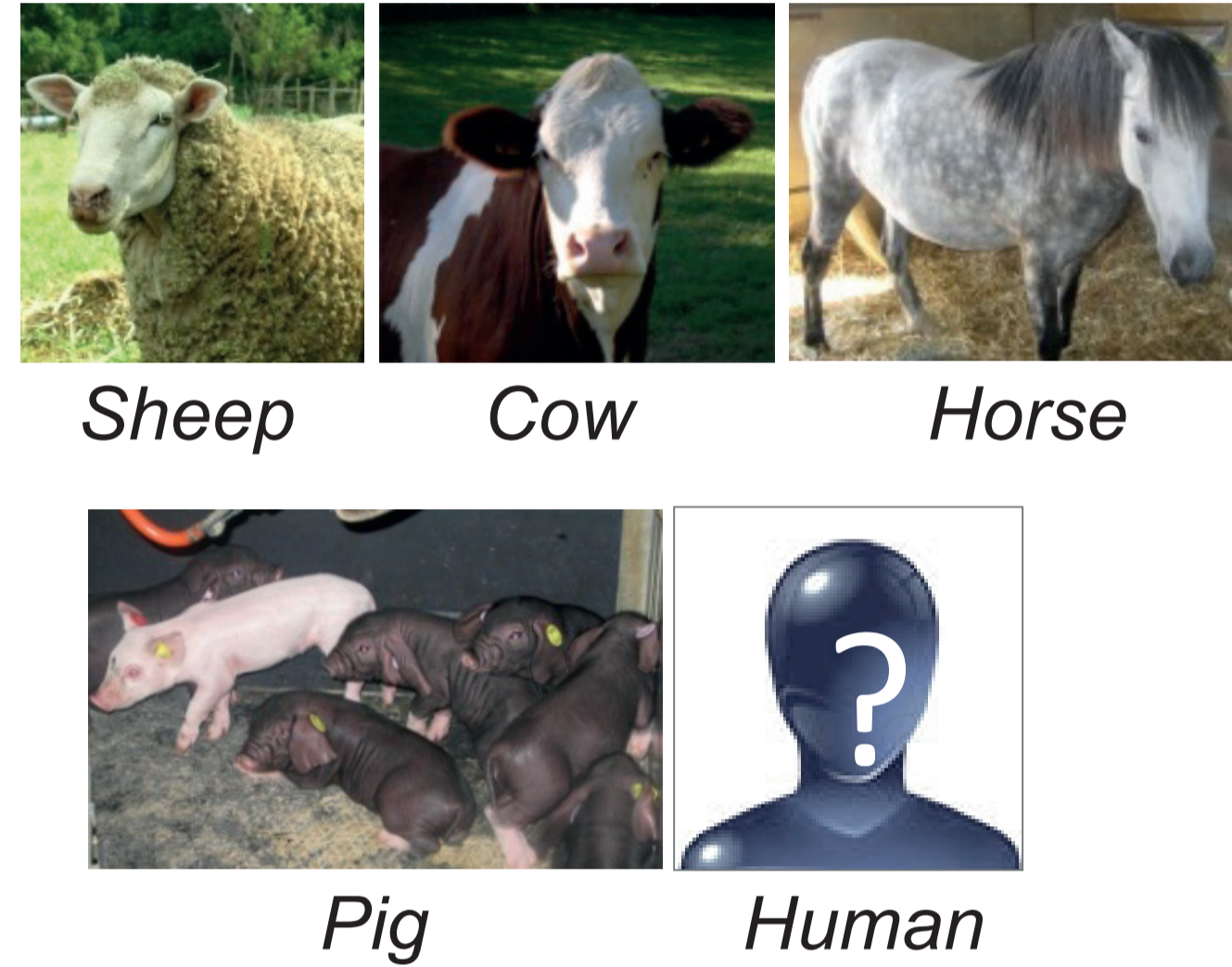
GC-MS details for sterols

| compound | MM | major m/z |
|----------|-----|--------------------|
| Cp | 388 | 215, 257, 355, 370 |
| epi-Cp | 388 | 215, 257, 355, 370 |
| Chl | 386 | 368, 353, 329, 255 |
| Cln | 388 | 215, 355, 455, 460 |
| mCp | 402 | 215, 257, 369, 384 |
| eCp | 416 | 215, 257, 383, 398 |
| epi-eCp | 416 | 215, 257, 383, 398 |
| Sterl | 412 | 255, 394, 379 |
| Stanl | 416 | 215, 257, 383, 398 |
| Sit | 414 | 396, 357, 381, 255 |

GC-MS details for bile acids

| compound | MM | major m/z |
|----------|-----|--------------------|
| LC | 462 | 215, 257, 357, 372 |
| DOC | 550 | 208, 255, 345, 370 |
| C | 638 | 253, 343, 368, 458 |
| CDOC | 550 | 255, 355, 370, 460 |
| HDOC | 550 | 255, 355, 370, 460 |
| UDOC | 550 | 255, 355, 370, 460 |

Reference Samples

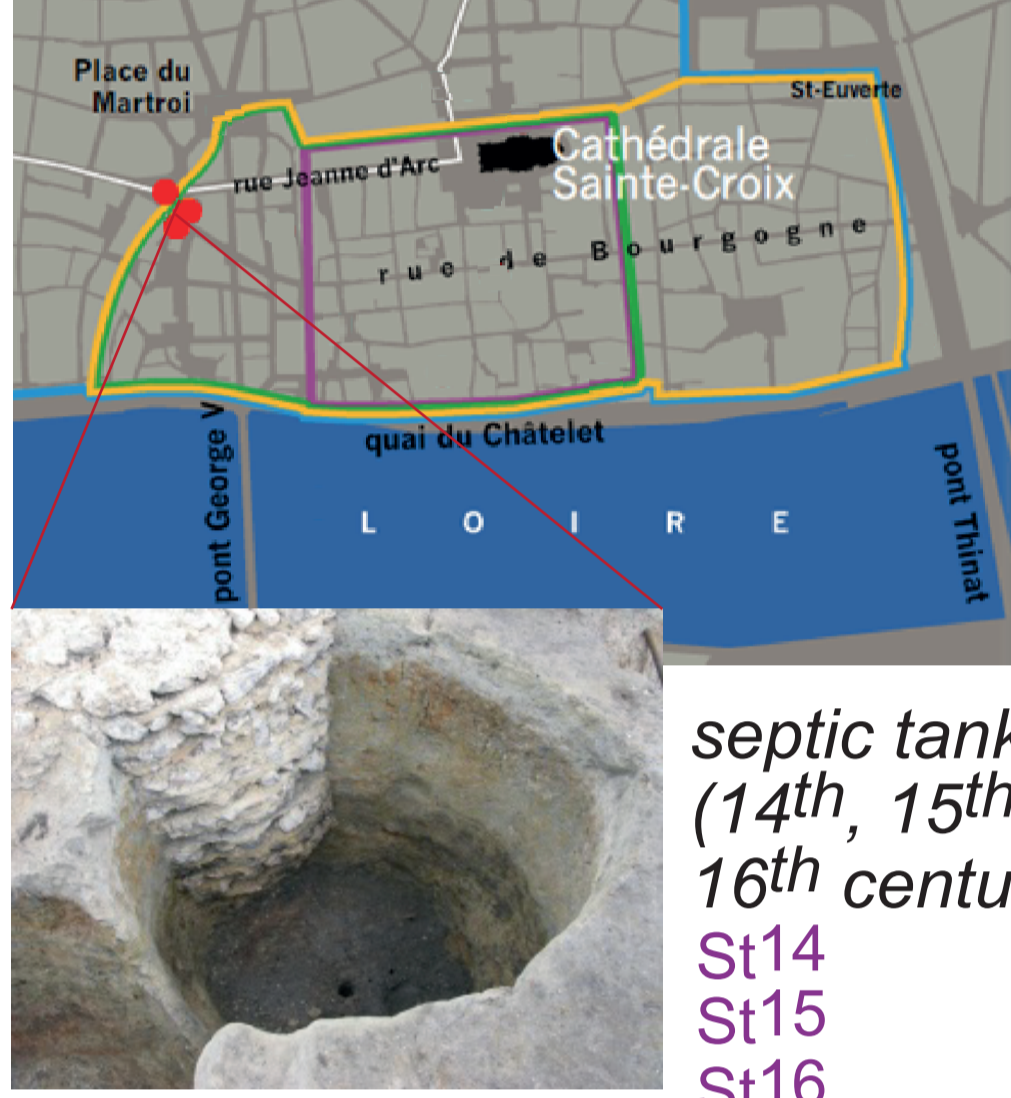


Collection Samples

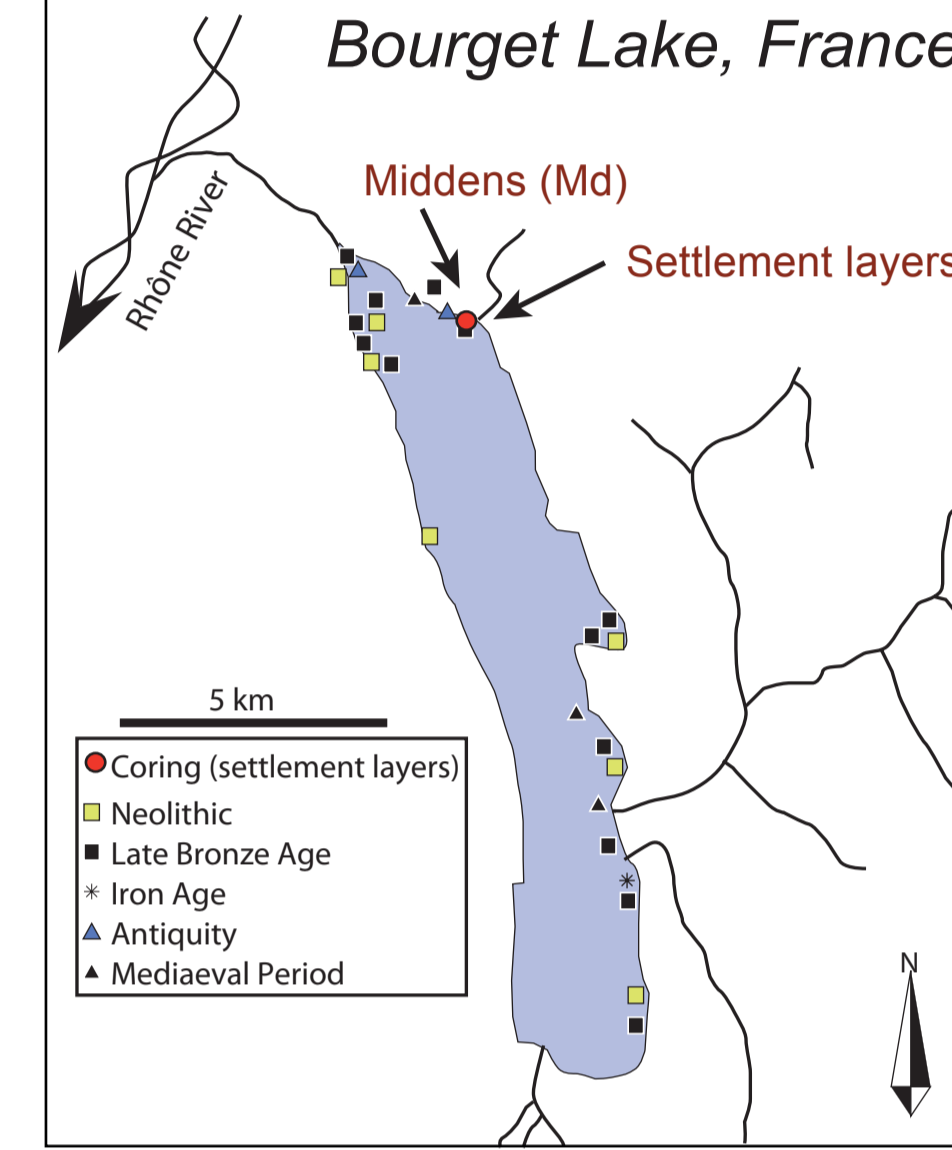
Septic tank



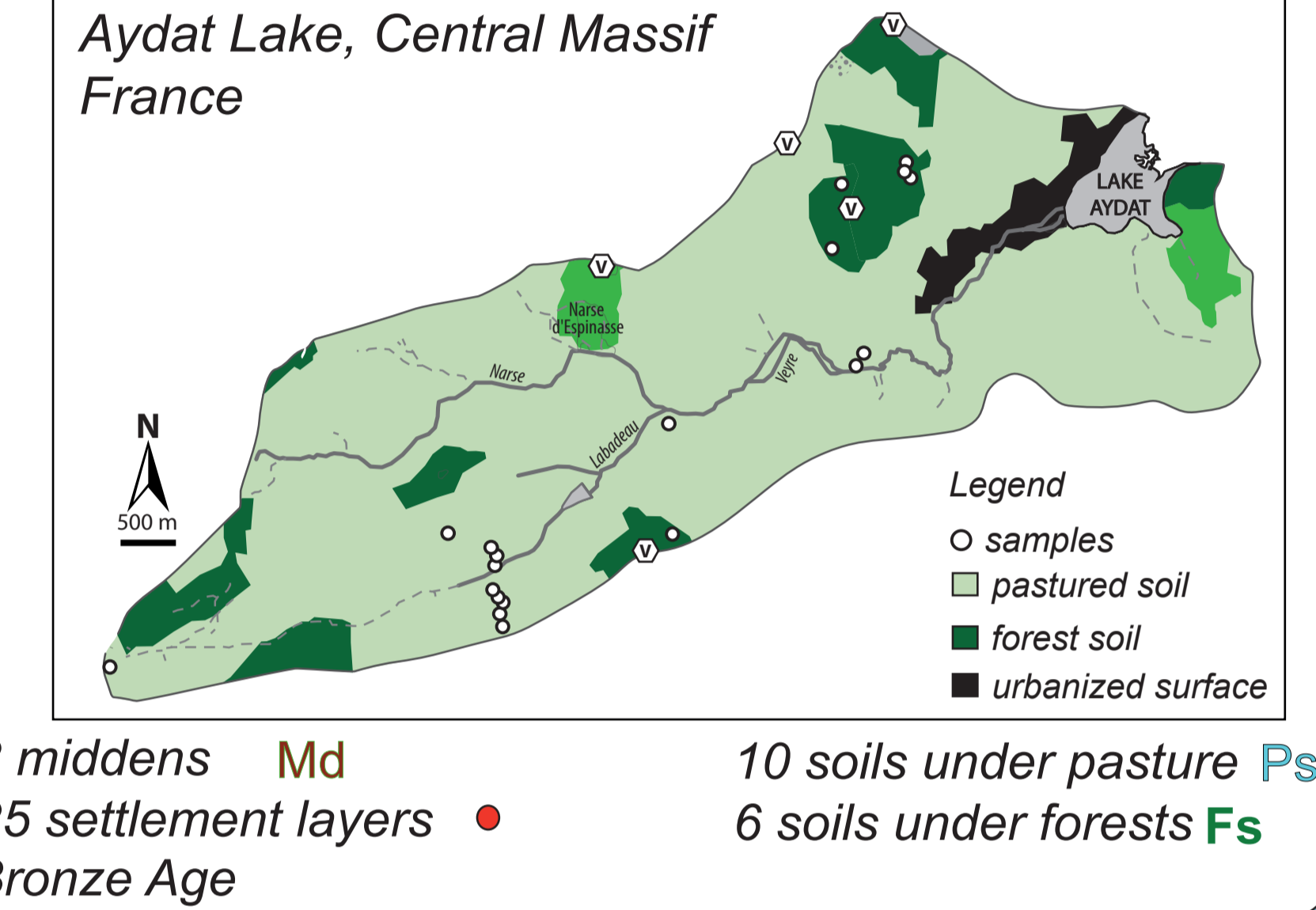
Archaeological excavations



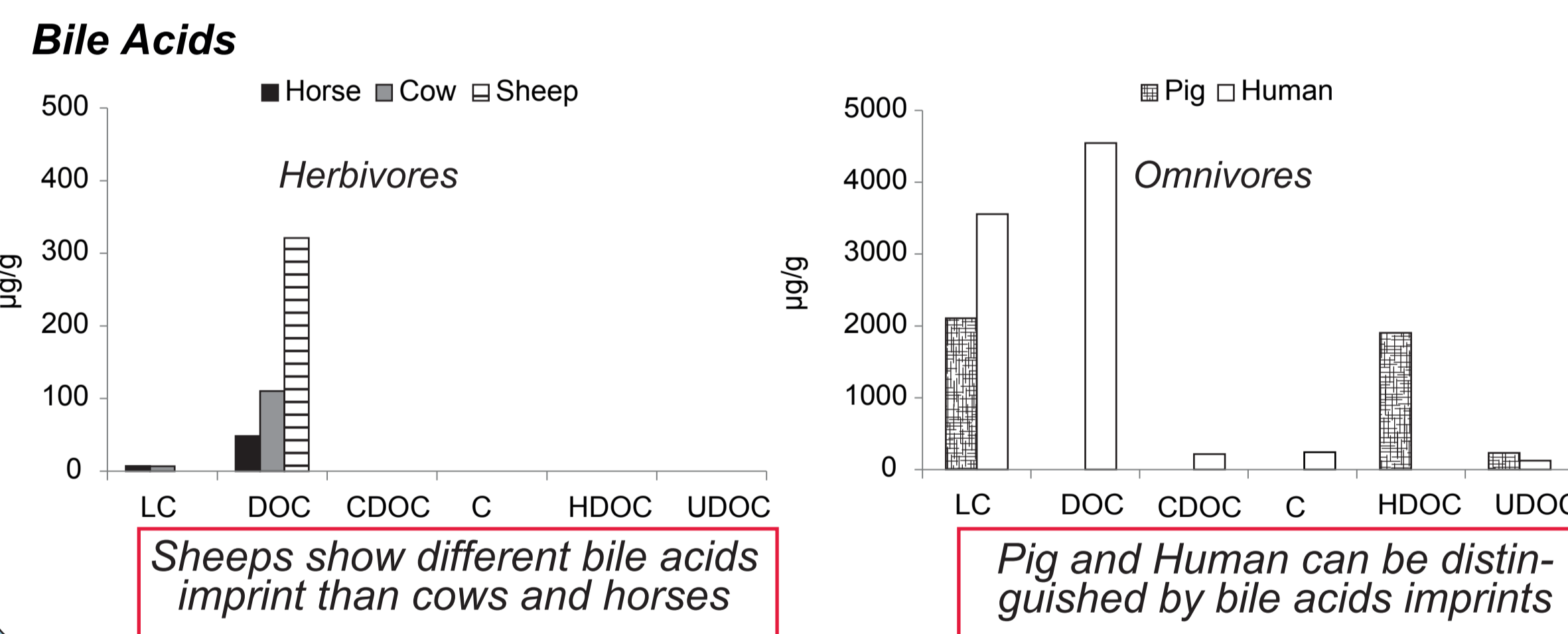
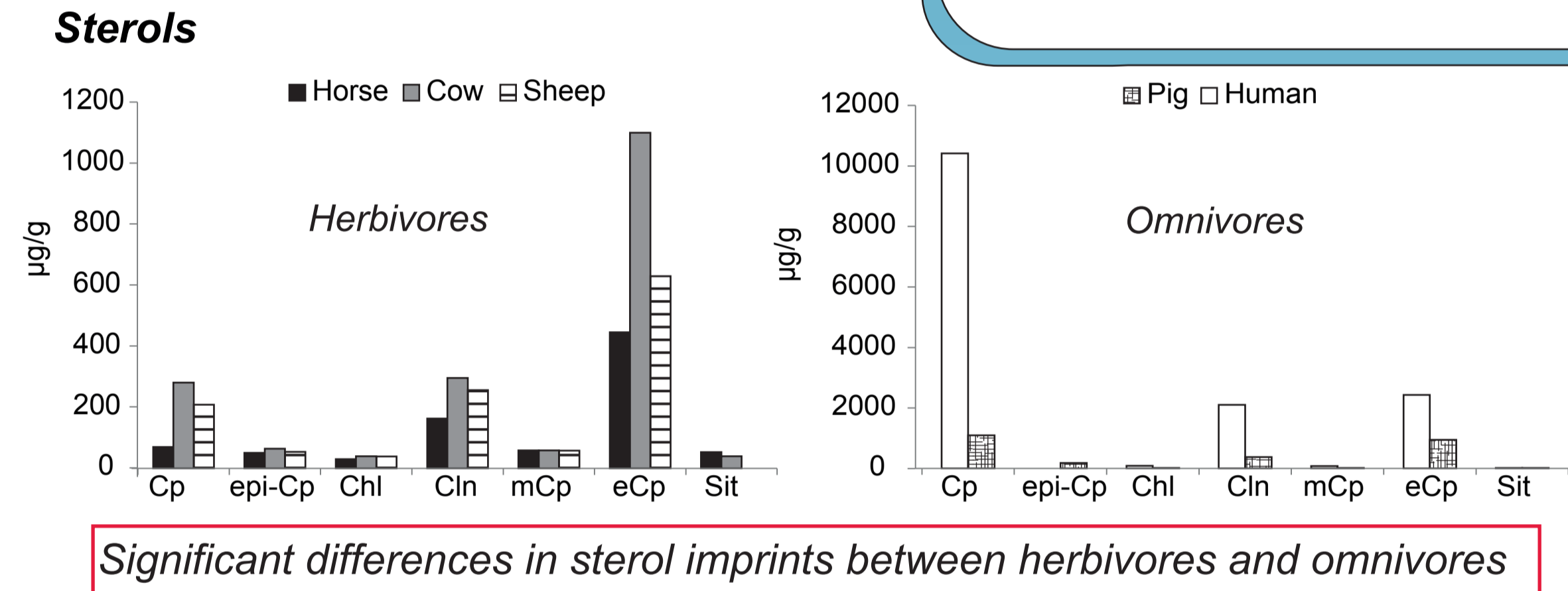
Archaeological site



Land-use Soils



Animal imprints



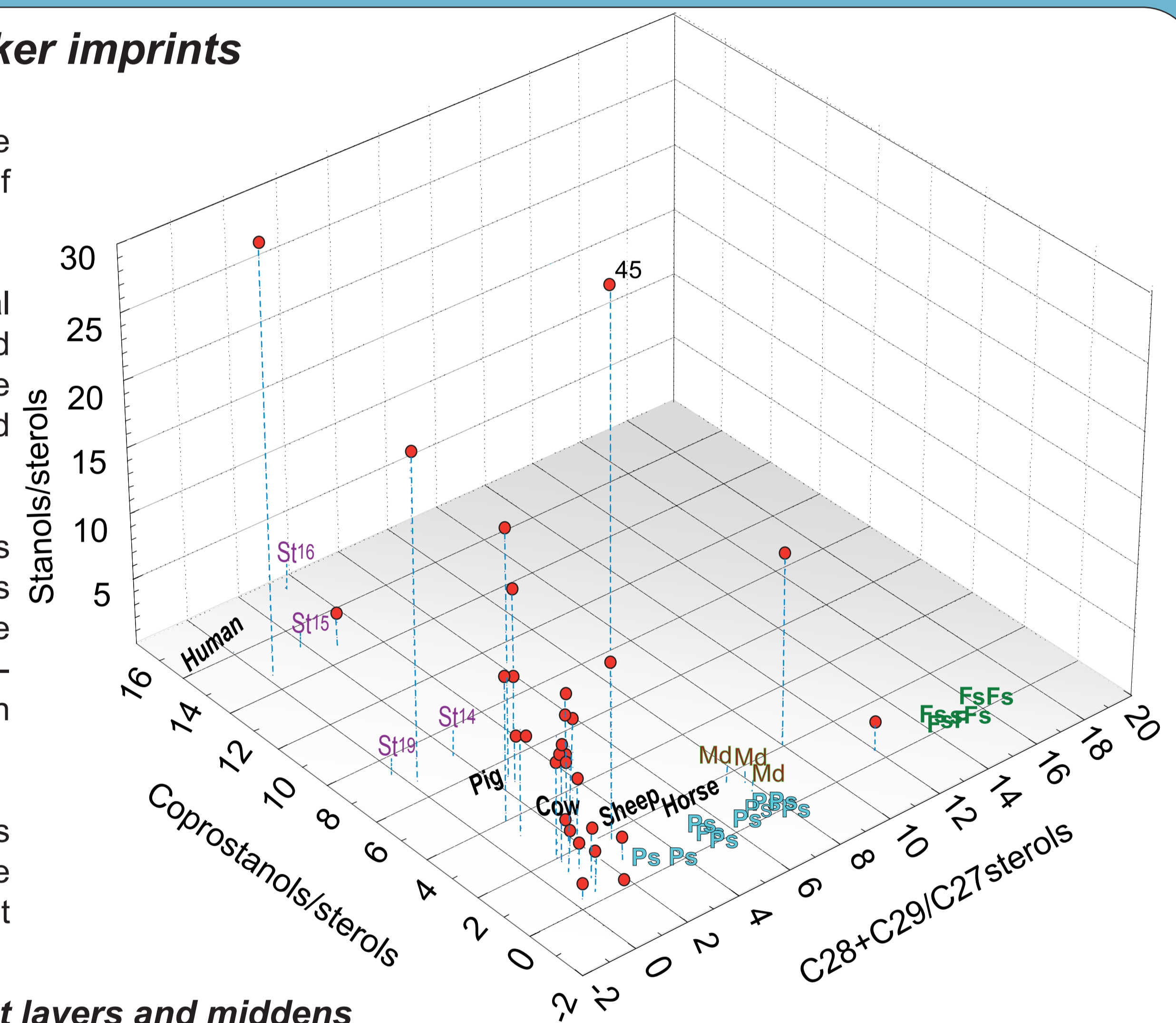
Variability of faecal biomarker imprints

Sterols index
Sterol ratios were used to identify the likely source and degradation state of faecal biomarkers in samples.

Recent samples such as the fresh faecal material (Reference samples), pastured and forest soils (Aydat catchment) are spread along coprostanol/sterol and C28+C29/C27 sterols axis.

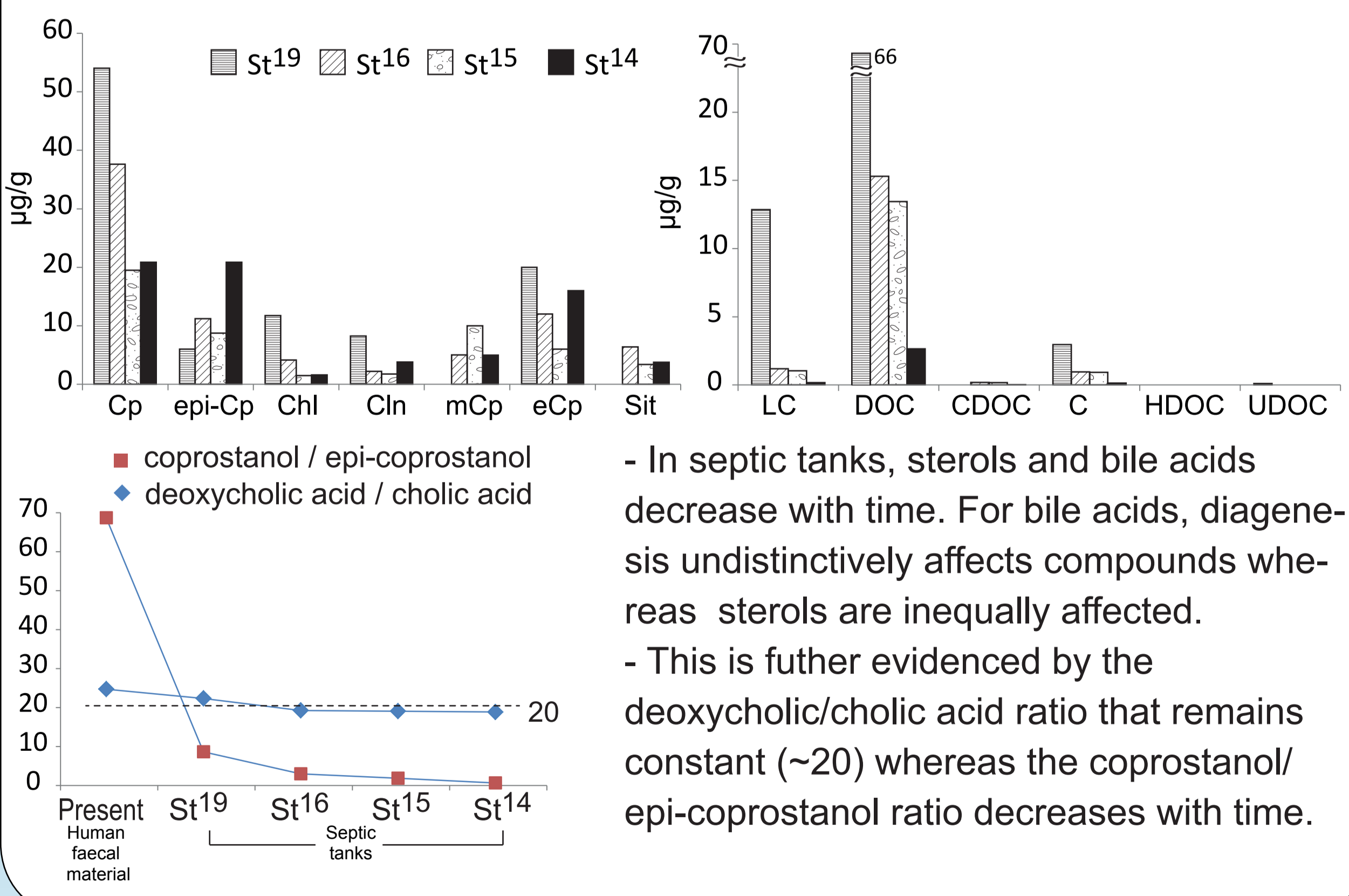
Archeological samples from septic tanks and middens have low stanols/sterols index whereas settlement layers have high values, indicating the sterols transformation into stanols (reduction) through time.

Diagenesis could entail coprostanols reduction thus masking the source imprints (Mermoud et al., 1984; Rushdi et al., 2006).

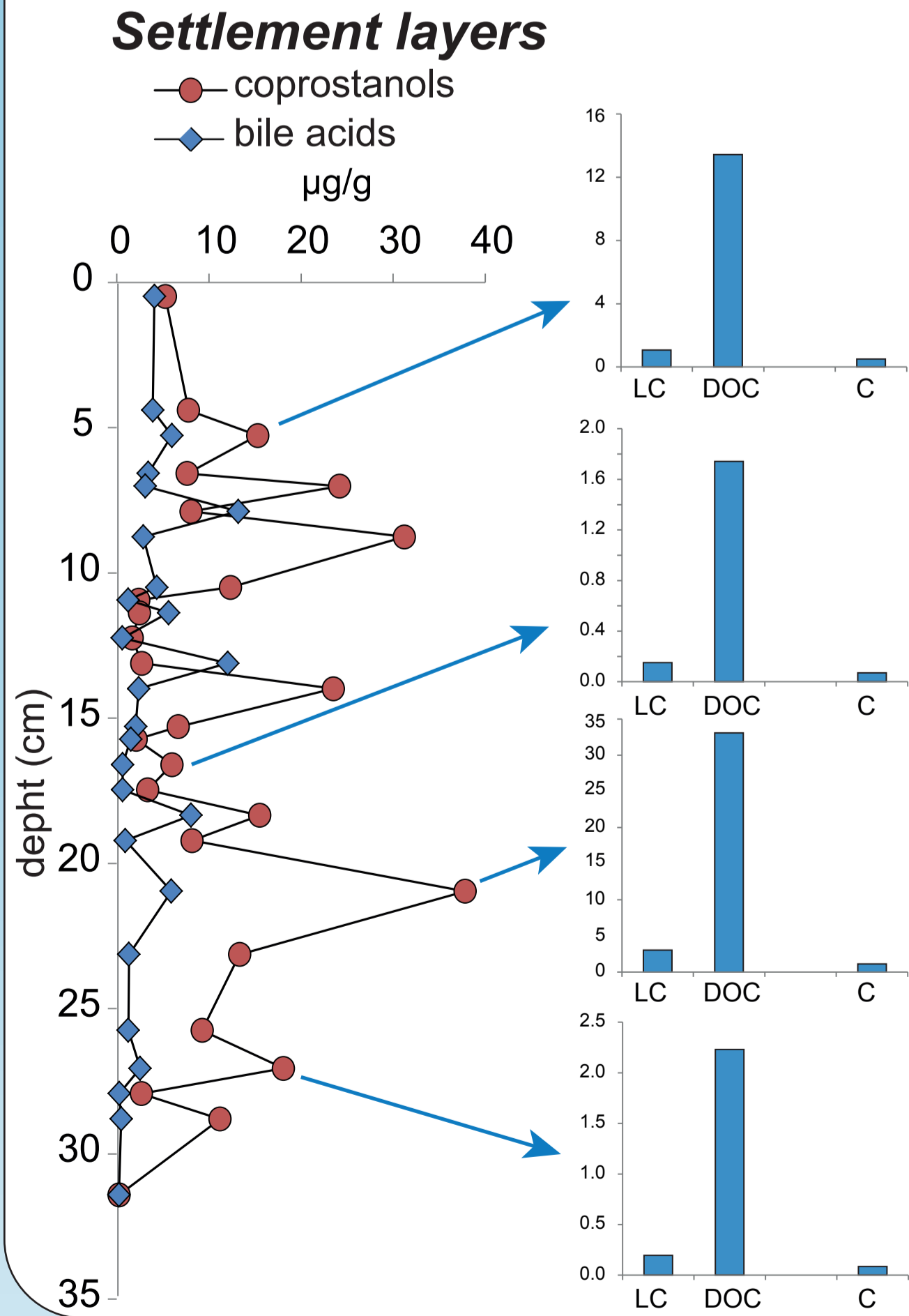


Faecal biomarkers preservation in septic tanks

Once demonstrated that animals have different imprints, we are interested in evaluating the preservation of faecal biomarkers in sedimentary profiles. Their distribution in septic tanks active during the 19th, 16th, 15th and 14th centuries are compared (St¹⁹, St¹⁶, St¹⁵ and St¹⁴, respectively).



Bile acids in Bronze Age settlement layers and middens



Samples from middens contain low amounts of bile acids (only LCA and DCA) indicative of cattle breeding. These data corroborate information obtained sterol data (in the same range as pastures, cows, sheeps and horses).

Coprostanols and bile acids vary across the profile in settlement layers. However, all settlement layers exhibit similar bile acids distribution with a deoxycholic/cholic acid ratio are greater than 20. This is indicative of a major contribution from Human feces but is contradictory to information retrieved from sterols that point to a significant contribution of cattle (cf. 3d graph). Nevertheless, sterols are more prone to degradation that can bias the original imprint.

Conclusions

- Significant differences in faecal biomarker imprints between allow distinguishing between animal sources: sheeps, cows, horses, pigs and Humans.
- Discrimination based on presence/absence of bile acids appear more effective than that based on sterols relative abundances.
- Bile acids proportion remain constant with time, and more stable than sterols.
- Combination of sterols and bile acids over a wide range contexts demonstrate their complementarity and efficiency in providing information on former land-uses.

Acknowledgments:

The study was accomplished as part of Otarie project (Region Centre and FEDER), ERODE project (EC2CO/CNRS/INSU) and PalHydroMil project (ANR). We thank Yves Billaud (DRASSM), Yohann Gerard (ISTO), Marlène Lavrieux (LSCE / Chrono-Environnement), Pascal Joyeux (INRAP) and Thomas Guillemard (INRAP) for their help during field works and for providing samples.

References:

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- Elhmmali et al., 2000. Environ. Sci. Technol. 34 (1), 39 - 46
- Bull et al., 2002. Environment International 27, 647- 654
- Jacob et al., 2005. Organic Geochemistry 36, 449 - 461
- Mermoud et al., 1984. Organic Geochemistry 6, 25 - 29
- Rushdi et al., 2006. Environmental Geology 50, 1171 - 1181

Bile acids in soils under pastures and forests

Soils under pastures contain notable amounts of lithocholic and deoxycholic acids that testifies to their present/recent use for cattle breeding or to manuring practises. Bile acids were absents in soils under forest.

