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Using portable energy dispersive X-ray fluorescence (EDXRF) analysis for on-site study of ceramic sherds at El Hibeh, Egypt

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Abstract

As part of its geoarchaeological research program, the El Hibeh Project of the University of California, Berkeley field-tested at the site of El Hibeh, northern Middle Egypt, the utility of a portable EDXRF unit for obtaining geochemical analyses of pottery suitable for provenance and other ceramic classification studies. When the geochemical ceramic data from the field test are combined with basic petrographic analysis for the pottery sample set, compelling results are generated. In particular, a triangular scattergram using elemental iron, strontium and rubidium data, in parts per million, provides excellent temporal and spatial separation of ceramic fabrics type, origin and date. The geochemical behavior of iron, strontium and rubidium is discussed in relation to geological source materials utilized for pottery manufacture.

Keywords: Pottery provenance; Portable EDXRF; El Hibeh project; Middle Egypt; Rb-Fe-Sr scattergrams; Egypt; Ceramic sourcing; Oasis ware

1. Introduction

As part of its on-going research program, the El Hibeh Project of the University of California, Berkeley is experimenting with a variety of methods for geoarchaeological field data collection and analysis. This paper focuses on ceramic sourcing techniques, a continuing concern of archaeologists and ceramic specialists working in Egypt and elsewhere. It asks three interrelated questions: 1) Can meaningful geochemical signatures be obtained from Egyptian ceramics that, with the aid of optical petrography, will be discriminatory for provenance, and also time period, and clay type?; 2) Can these discriminatory geochemical signatures be obtained using a portable geochemical system that is suitable for field use?; and 3) Can this signature identify locally produced Nile Valley ceramics, and, possibly in the future, pottery manufactured at specific archaeological sites such as El Hibeh?

2. Archaeological context

El Hibeh lies on the east bank of the Nile River in northern Middle Egypt, approximately 55 km south of the modern governorate capital of Beni Suef and 165 km south of Cairo, Egypt's capital (Fig. 1). This multi-component site includes the remnants of a walled settlement, once a provincial town, as well as necropoleis of different dates that extend into the adjacent desert around the tell. Inside the massive mudbrick town walls that today surround the site on three sides (the fourth,

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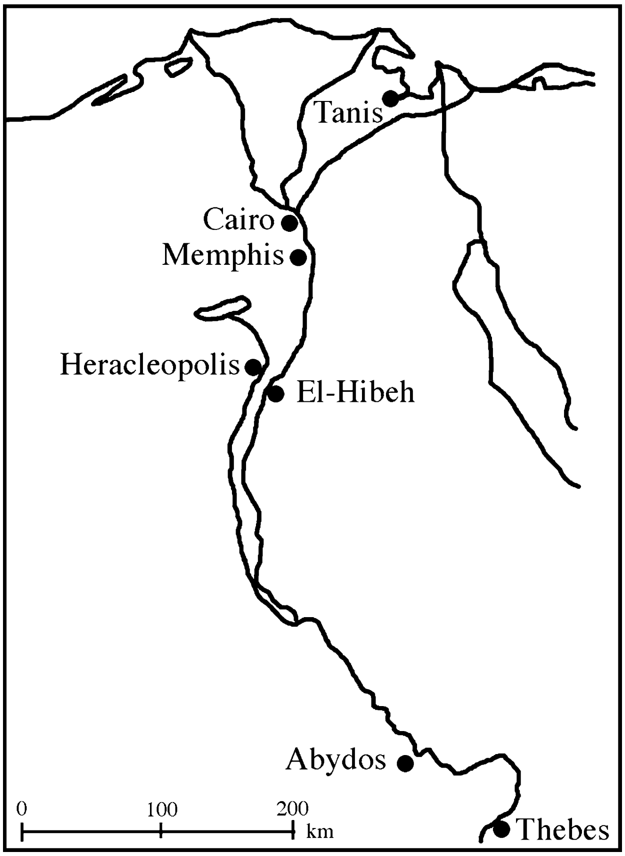

Fig. 1. Location of El-Hibeh.

western side fronted on or was in close proximity to the Nile River) are a small limestone temple, dated by inscriptions to the tenth century BCE and lying within its own mudbrick temenos wall; industrial debris; habitation remains; and mortuary deposits. Present evidence, both archaeological and textual, indicates that the town was founded at the beginning of Egypt's Third Intermediate period (TIP; early eleventh century B.C.E.) and was occupied, more or less continually, until at least as late as the fourth or fifth century C.E.

The pottery used in this test study was selected from a collection of surface survey ceramics. The majority of these ceramics came from surface clearance samples gathered near a mortuary deposit, Burial Cave 1 (BC-1), located in the central northern part of the tell. The remainder was obtained from various locations throughout the mound. In future, as we expand and refine our work, we will concentrate on material with clearer archaeological contexts. At present, we have focused on developing a technique for field provenance studies in Egypt.

3. Methods

A total of forty-seven surface survey sherds (Table 1) were chosen for analysis based on ease of general visual identification and suitable size (see below). Visual assessment and petrographic analysis were completed in the field on each sherd. A freshly broken edge was used for observations; no thin sections were made. Petrographic analysis was undertaken using a Nikon binocular stereomicroscope. The following basic data were collected for each sample: general vessel type and date; basic fabric type; basic type(s) of temper or inclusions or both; very general pore characteristics; and Munsell color as appropriate for exterior surface, interior surface, core rind (outer part of core) and core.

Optical petrography and geochemical analysis are two complementary techniques that should, in our view, be used together whenever possible for optimal understanding of a given ceramic set. Petrographic analysis identifies, inter alia, the fundamental mineralogic character of a sample—whether, for example, it is composed of a marl or silt fabric. It therefore can be critical for guiding the correct use of comparative geochemical data. It is inappropriate, for example, to compare the geochemical signatures of marl clays with those of Nile silts, as there is no geochemical reason they should correlate. Marls should be compared geochemically to other marls and silts to other silts, and so forth.

Geochemical data were collected on the samples using a NITON XLt-793W portable EDXRF (energy dispersive X-ray fluorescence) spectrometer (Fig. 2) that produced measurements in parts per million for a total of seventeen elements (Table 2). The NITON Xlt X-ray tube-based analyzer is completely portable. It has one hand trigger operation, a touch screen with full navigation, complete energy spectra view and scanning, and digital screen printout with an RS232 download port. We operated it in bulk sample mode using the complete list of element concentration and detection limits on the results screen. The unit saves all data (up to 3000 readings) until prompted to erase. Our unit has two rechargeable 12-volt batteries that give us more than a full day of field use, and are easily recharged in Egypt. All geochemical analyses were carried out in the field. In order to determine optimal exposure times for the study samples, powdered standards and test pottery sherds were first subjected to analysis by the unit for exposure times ranging from 60 to 400 seconds. Detection-limit data then were plotted against time of exposure to determine where the detection-limit curves changed slope. Past this inflection point, which was found to be at 240 seconds, additional exposure time did not markedly improve detection limits. All samples in this study therefore were exposed for a minimum of 240 seconds. All tested samples were larger than the NITON's exposure window, which measures 1 cm × 2 cm. The flattest portion of each ceramic sherd sample was placed next to the exposure window for analysis. Calibration standards were used prior to analysis.

The NITON XLt-793W portable EDXRF was chosen not only because it could be used in the field

Table 1
Fabric and ware type for EH04 surface pottery sherds

Sample no.	Type and age	Fabric paste	Temper (inclusions)	Pore	Exterior color	Core color	Core rind	Interior color
C1	Roman amphora base	Nile silt	Organics phytoliths	Few	10YR5/4	10R5/1	N/A	10YR5/4
C2	TIP jar	Nile silt	Quartz sand organics	Large round	10R4/6	10R4/6	N/A	10R4/6
C3	TIP jar rim	Nile silt	Organics phytoliths	Large long	2.5YR4/2	2.5YR4/3	5YR6/2	2.5YR4/3
C4	TIP jar	Nile silt	Limestone granules & quartz sand	Round	10R5/4	10YR5/4	N/A	10YR5/4
C5	Hellenistic large bowl rim	Nile silt	Organics	Large long	2.5YR5/4	5YR4/1	N/A	2.5YR5/4
C6	Hellenistic krater rim	Nile silt	Organics	Large long	10R5/4	7.5YR4/1	N/A	10R5/4
C7	ARS ware bowl import	Marl	(Fine to very fine quartz sand)	Few	10R5/6	10R5/6	N/A	10R5/6
C8	ARS ware bowl import	Marl	(Fine to very fine quartz sand)	Few	10R5/6	10R5/6	N/A	10R5/6
C9	ARS ware bowl import	Marl	(Fine to very fine quartz sand)	Few	10R5/6	10R5/6	N/A	10R5/6
C10	Coptic painted jar	Nile silt	Organics & limestone granules	Large long	2.5YR4/6	N/A	N/A	2.5YR4/1
C11	Modern	Marl	Ash	Few	2.5YR6/6	N/A	N/A	2.5Y6/3
C12	Oasis ware	Silt	Quartz sand & ash, organics & (carbonate blows*)	Round	10YR7/6	2.5Y6/1	N/A	5YR8/6
C13	Oasis ware	Silt	Quartz sand	Round	2.5Y5/2	7.5YR6/4	N/A	2.5Y5/2
C14	Oasis ware	Silt	Quartz sand & ash, organics & (carbonate blows*)	Round	5/YR4/1	2.5YR6/2	10YR7/6 & 2.5YR6/4	7.5YR6/3
C15	Oasis ware	Silt	Quartz sand & ash, organics & (carbonate blows*)	Round	7.5YR5/2	2.5Y5/2	N/A	2.5Y5/2
C16	Oasis ware	Silt	Quartz sand & ash, organics & (carbonate blows*)	Round	10YR4/1	10YR5/1	10YR6/4	10YR4/1
C17	Oasis ware	Silt	Quartz sand (carbonate blows*)	Round	2.5YR6/4	10YR5/1	N/A	10YR6/4
C18	Oasis ware	Silt	Quartz sand	Round	10YR4/1	2.5YR6/2	7.5YR6/6 & 5YR6/6	2.5YR6/2
C19	TIP striped ware	Silt	Carbonate blows* major organics	Few round	2.5YR5/6	10YR5/3	N/A	2.5Y6/2
C20	TIP striped ware	Silt	Carbonate blows* organics	Round	2.5YR6/4	2.5YR5/4	N/A	2.5YR4/3
C21	TIP striped ware	Silt	Carbonate blows* minor organics phytoliths	Large round	2.5YR4/4	2.5YR5/6	N/A	7.5YR4/2
C22	Greek amphora import	Marl	Limestone fragments (minor quartz silt)	Few	2.5YR7/3	2.5YR5/6	N/A	2.5YR7/3
C23	Greek amphora import	Marl	Limestone fragments (some quartz silt)	Many	5Y7/3	2.5YR6/4	N/A	5Y7/3
C24	Greek amphora import	Marl	Limestone fragments (lots of quartz silt)	Few	2.5YR7/3	5YR6/4	N/A	5YR6/6
C25	Hellenistic/roman cookpot	Nile silt	None	Very few	10R5/6	2.5YR5/1	N/A	10R5/6
C26	Hellenistic/roman cookpot	Nile silt	Quartz sand rounded and polished	Few	10R5/4	10R4/1	N/A	10R5/4
C27	Hellenistic/roman cookpot	Nile silt	Quartz and carbonate sand	Few	ND	ND	ND	ND
C28	Hellenistic/roman cookpot	Nile silt	(Carbonate blows)	Few	ND	ND	ND	ND
C29	Hellenistic/roman cookpot	Nile silt	Quartz sand rounded and polished	Few	10R5/4	10R4/1	N/A	10R5/4
C30	Hellenistic/roman cookpot	Nile silt	Limestone granules	Few	10R5/6	2.5YR5/1	N/A	10R5/6
C31	Hellenistic/roman cookpot	Nile silt	None	Very few	10R5/4	10R4/1	N/A	10R5/4

(*continued*)

Table 1 (*continued*)

Sample no.	Type and age	Fabric paste	Temper (inclusions)	Pore	Exterior color	Core color	Core rind	Interior color
C32	Hellenistic/roman cookpot	Nile silt	Quartz sand rounded and polished (carbonate blows*)	Few	10R5/4	N/A	N/A	10R5/4
C33	Hellenistic/roman cookpot	Nile silt	(Quartz coarse silt)	Very few	ND	ND	ND	ND
C34	Hellenistic/roman casserole	Nile silt	Some fine to medium quartz sand	Few	ND	ND	ND	ND
C35	Hellenistic/roman casserole	Nile silt	Quartz sand (very few large organic voids)	Very few large long	ND	ND	ND	ND
C36	Hellenistic/roman casserole	Nile silt	Abundant quartz sand Rounded and polished	Few	ND	ND	ND	ND
C37	TIP baking tray	Nile silt	Organics phytoliths, Limestone sand	Long	2.5YR5/4	2.5Y4/1	N/A	2.5YR5/4
C38	TIP baking tray	Nile silt	Organics phytoliths, Limestone sand	Long	2.5YR5/4	2.5Y4/1	N/A	2.5YR5/4
C39	TIP baking tray	Nile Silt	Organics phytoliths, Limestone sand	Long	2.5YR5/4	2.5Y4/1	N/A	2.5YR5/4
C40	TIP baking tray	Nile silt	Organics phytoliths, Limestone Sand	Long	2.5YR5/4	2.5Y4/1	N/A	2.5YR5/4
C41	Basin	Nile silt	Organics phytoliths, Limestone sand	Long	2.5YR5/4 2.5YR5/6	2.5Y4/1 7.5YR4/1	N/A	2.5YR5/ 6.5YR5/4
C42	Basin	Nile silt	Organics phytoliths Limestone sand	Long	ND	ND	ND	ND
C43	Islamic silt bowl, glazed	Silt	Quartz sand	Small	Gley1: 2.5/N	5YR2.5/1 2.5Y4/6	N/A	Glaze N/A
C44	Green marl	Marl	Ash organics (quartz silt)	Long	5Y6/3	5Y6/3	N/A	5Y7/3
C45	Late/coptic surface painted red-black, white slipped	Silt	Organics, carbonate blows*	Many long	2.5YR5/6	2.5Y5/2	N/A	2.5Y5/2
C46	Jug rim w/handle & high filter	Marl	Quartz sand ash	Few sub-round	2.5Y7/3	5YR6/4	N/A	5YR6/4
C47	Fine small jar base	Marl	Quartz silt (carbonate blows*)	Sub-round	5YR6/4	10YR6/3	N/A	10YR6/3

* Carbonate blows are calcium oxide coated pores that were calcite/limestone grains prior to firing the ceramic.

but also because it is an X-ray tube model that does not have a radioactive source and therefore may travel freely to different countries. An earlier attempt to bring into Egypt for field use a portable EDXRF NITON 703S unit with a sealed cadmium source (10 mCi Cd-109) configured to handle 15 elements failed when the Egyptian Ministry of Health refused to give permission for the unit to enter the country. The seventeen elements offered on the NITON XLt-793W portable unit are not ideal for ceramic provenance work as some key elements normally used for such work, such as scandium, lanthanum, thorium and uranium, are not easily available with low detection limits and at least six of the seventeen elements that are available are found in natural sediments at levels below the normal portable spectrometer detection levels. Nevertheless, the remaining eleven elements proved to be quite adequate for our sourcing purposes.

Fig. 2. NITON Xlt 793W portable EDXRF.

Of the seventeen elements analyzed by the NITON, eleven had high enough values over detection limits to be useful: Sb, Sn, Ag, Sr, Rb, Pb, Zn, Cu, Co, Fe, and Mn. We used these eleven elements for a hierarchical cluster analysis created by SPSS-11 statistical software. This analysis produced a dendogram of the sample sherds showing the linkages between groups (Fig. 3). This analysis discriminated Nile Valley (Nile River floodplain sediments and bordering desert outcrops) from non-local ware types and was useful for this purpose only. We define the term Nile Valley ware, for the purposed of this paper, as all fabrics (marls and silts) deriving from Nile River floodplain sediments and bordering desert outcrops and sediments. We define none local wares as all

Table 2
NITON Xlt-793W portable EDXRF ceramic sherd geochemistry in parts per million (ppm)

#	Sb	Sn	Cd	Ag	Sr	Rb	Pb	Se	As	Hg	Zn	Cu	Ni	Co	Fe	Mn	Cr
C1	136.3±61.8	88.8±57.6	Bdl	31.8±23.5	268.7±8.3	36.0±3.8	14.7±9.6	Bdl	Bdl	Bdl	Bdl	83.3±44.7	Bdl	438.3±173.5	52,600±500	669.8±109.4	Bdl
C2	204.8±59.0	132.2±54.8	61.6±25.4	43.2±22.3	271.9±8.0	44.3±3.8	16.2±9.1	Bdl	Bdl	Bdl	70.4±22.1	82.4±41.7	Bdl	324.0±165.0	51,900±500	1000±100	Bdl
C3	204.9±58.2	122.7±54.0	55.3±25.0	35.1±21.9	304.0±8.3	42.8±3.8	13.1±8.9	Bdl	Bdl	Bdl	54.9±21.4	83.6±40.9	Bdl	223.6±169.4	56,700±500	789.1±107.8	Bdl
C4	182.9±61.0	87.1±56.5	Bdl	34.3±23.0	413.6±9.9	40.9±3.9	12.4±9.3	Bdl	Bdl	Bdl	Bdl	100.3±43.9	Bdl	462.6±160.4	45,200±400	1100±100	Bdl
C5	182.9±56.7	80.1±52.6	Bdl	34.6±21.4	385.1±9.1	41.6±3.7	35.8±9.7	Bdl	9.4±7.3	Bdl	61.7±21.0	109.4±40.2	Bdl	520.8±172.7	59,100±500	15001±100	131.6±94.9
C6	210.5±56.7	Bdl	38.6±24.3	37.5±21.4	337.5±8.6	38.3±3.6	11.3±8.6	Bdl	Bdl	Bdl	41.4±20.4	65.3±39.2	Bdl	296.7±172.2	60,800±500	1000±100	Bdl
C7	137.1±42.2	64.7±39.1	Bdl	27.0±15.9	305.3±6.6	92.8±3.9	18.7±6.6	Bdl	Bdl	Bdl	45.0±13.9	43.4±25.9	Bdl	114.0±96.7	28,300±300	269.4±61.4	79.6±60.9
C8	126.9±43.5	59.6±40.3	Bdl	Bdl	195.9±5.6	72.1±3.6	13.2±6.5	Bdl	8.3±5.0	Bdl	31.5±14.1	48.0±27.2	Bdl	Bdl	28,100±300	Bdl	Bdl
C9	169.1±43.5	86.6±40.3	Bdl	25.4±16.4	410.7±7.7	98.5±4.1	15.0±6.7	Bdl	12.5±5.2	Bdl	25.7±13.9	Bdl	Bdl	Bdl	29,700±300	200.1±60.9	Bdl
C10	143.5±55.0	60.4±51.1	Bdl	34.0±20.9	307.7±8.1	39.4±3.5	9.5±8.2	Bdl	Bdl	Bdl	Bdl	86.2±38.5	Bdl	311.1±170.7	62,200±500	1200±100	116.6±92.4
C11	117.7±54.4	Bdl	Bdl	32.6±20.6	538.3±10.3	48.7±3.8	23.3±8.8	Bdl	Bdl	Bdl	119.5±21.3	77.9±37.9	Bdl	Bdl	37,100±400	297.8±82.9	151.6±85.9
C12	124.3±45.9	88.1±42.6	Bdl	19.7±17.3	154.5±5.3	26.1±2.7	9.6±6.7	Bdl	Bdl	Bdl	48.0±15.4	37.9±29.1	37.8±26.8	86.2±84.6	18,400±200	407.0±67.5	Bdl
C13	158.9±43.6	57.9±40.3	Bdl	28.8±16.4	180.3±5.4	45.0±3.0	15.7±6.6	Bdl	5.1±4.9	Bdl	49.9±14.4	54.7±27.3	42.5±25.5	Bdl	20,600±200	745.2±74.7	Bdl
C14	139.6±44.5	93.9±41.3	Bdl	Bdl	132.6±4.9	38.8±2.9	19.9±6.9	Bdl	Bdl	Bdl	41.0±14.6	73.6±28.6	Bdl	109.6±86.5	20,200±200	511.3±64.7	81.0±62.3
C15	142.7±46.1	Bdl	Bdl	22.0±17.4	329.2±7.3	42.1±3.1	24.3±7.4	Bdl	9.5±5.6	Bdl	63.9±15.9	76.0±30.1	52.0±27.5	106.4±85.8	18,600±200	2700±100	119.7±66.5
C16	130.0±44.2	50.7±40.9	Bdl	24.4±16.7	208.4±5.8	44.6±3.0	41.5±7.7	Bdl	16.6±6.1	Bdl	34.4±14.3	70.4±28.2	Bdl	Bdl	20,200±200	2000±100	Bdl
C17	130.6±44.7	78.7±41.5	20.0±19.2	Bdl	263.6±6.4	46.6±3.1	27.6±7.3	Bdl	Bdl	Bdl	53.6±15.2	57.7±28.8	Bdl	112.7±86.4	20,200±200	525.7±70.4	86.7±63.0
C18	178.5±46.1	86.7±42.7	22.3±19.7	45.0±17.5	357.5±7.5	42.6±3.1	39.9±8.0	Bdl	Bdl	Bdl	40.9±15.2	74.4±29.8	Bdl	111.8±88.3	20,000±200	1300±100	76.1±64.5
C19	137.5±52.3	Bdl	Bdl	33.6±19.8	419.8±8.9	45.9±3.6	10.0±7.9	Bdl	Bdl	Bdl	86.6±19.8	91.6±36.0	59.3±34.4	231.4±163.4	60,900±500	1200±100	131.0±88.3
C20	155.8±53.5	79.0±49.8	Bdl	44.0±20.3	342.4±8.3	41.0±3.5	19.1±8.5	Bdl	Bdl	Bdl	131.2±21.4	94.9±37.1	44.1±35.0	293.5±169.2	63,100±500	1300±100	Bdl
C21	108.8±49.5	Bdl	Bdl	23.2±18.7	360.2±8.0	37.1±3.2	15.5±7.6	Bdl	Bdl	Bdl	42.7±17.1	60.3±33.1	Bdl	510.6±123.9	34,500±300	782.9±89.2	Bdl
C22	192.9±50.5	120.1±46.9	26.1±21.6	52.6±19.1	301.4±7.5	143.3±5.3	54.6±9.4	Bdl	8.9±7.1	Bdl	40.4±17.3	72.5±33.9	211.6±35.8	Bdl	33,000±300	440.8±80.1	152.2±77.9
C23	79.1±52.0	65.2±48.5	Bdl	31.8±19.8	466.8±9.3	42.3±3.5	16.4±8.1	Bdl	Bdl	Bdl	54.5±18.7	90.6±36.3	40.5±32.3	Bdl	36,800±400	580.0±88.1	101.1±80.3
C24	78.8±50.0	Bdl	Bdl	Bdl	626.2±10.4	38.3±3.3	14.7±7.7	Bdl	6.5±5.8	Bdl	76.2±18.2	60.4±33.6	Bdl	142.5±113.1	29,700±300	393.8±77.2	Bdl
C25	165.4±54.1	Bdl	Bdl	41.1±20.5	340.4±8.3	42.5±3.6	13.4±8.3	Bdl	Bdl	Bdl	41.3±19.6	79.5±37.3	Bdl	492.0±185.3	74,100±500	1300±100	167.6±95.9
C26	162.7±57.0	Bdl	Bdl	Bdl	268.4±7.8	37.8±3.6	23.8±9.2	Bdl	13.1±7.1	Bdl	89.9±21.9	73.3±39.9	Bdl	358.2±174.8	61,200±500	1800±100	Bdl
C27	115.1±56.9	Bdl	Bdl	26.4±21.6	480.5±10.1	30.4±3.4	44.2±10.1	Bdl	Bdl	Bdl	5300±100	117.3±41.4	Bdl	303.2±157.1	48,300±400	1000±100	147.1±94.9
C28	257.8±59.2	122.4±54.9	30.0±25.2	48.2±22.3	371.2±9.2	41.0±3.8	17.2±9.3	Bdl	Bdl	Bdl	120.7±23.7	139.8±42.7	Bdl	469.8±195.1	73,300±500	1300±100	Bdl
C29	145.2±53.4	66.4±49.6	Bdl	Bdl	354.8±8.4	35.3±3.3	22.3±8.5	Bdl	Bdl	Bdl	38.0±18.9	88.5±37.0	37.3±34.0	223.4±154.8	52,800±400	1100±100	Bdl
C30	131.6±51.1	35.1±47.4	Bdl	Bdl	332.4±7.9	36.7±3.3	12.6±7.8	Bdl	Bdl	Bdl	35.0±18.1	107.6±35.2	42.7±33.7	361.9±168.8	66,600±500	1100±100	135.3±88.0
C31	143.6±57.2	Bdl	40.8±24.7	Bdl	426.6±9.6	39.9±3.7	14.4±8.9	Bdl	7.3±6.7	Bdl	48.4±21.1	104.6±40.7	Bdl	469.1±185.1	68,000±500	1100±100	97.4±97.0
C32	126.2±50.1	61.2±46.6	30.3±21.6	30.2±19.0	299.0±7.5	34.2±3.1	8.5±7.5	Bdl	7.7±5.7	Bdl	24.6±17.4	107.1±34.3	Bdl	334.6±164.5	65,100±500	1200±100	123.3±85.6
C33	92.7±57.5	Bdl	Bdl	22.9±21.8	326.7±8.6	40.5±3.7	18.5±9.2	Bdl	Bdl	Bdl	39.0±21.0	Bdl	Bdl	403.4±176.1	60,500±500	1300±100	97.7±95.9
C34	90.6±51.5	Bdl	Bdl	25.7±19.6	471.9±9.4	39.0±3.4	11.3±7.9	Bdl	Bdl	Bdl	49.2±18.6	63.5±35.0	Bdl	623.4±167.9	62,800±500	1300±100	Bdl
C35	156.4±56.2	66.7±52.2	Bdl	35.0±21.3	301.8±8.1	40.9±3.6	24.7±9.1	Bdl	Bdl	Bdl	37.7±20.2	126.3±40.2	Bdl	452.5±166.8	55,900±500	1100±100	105.5±92.2
C36	93.5±51.6	50.2±48.0	Bdl	Bdl	272.6±7.3	34.8±3.2	11.0±7.8	Bdl	Bdl	Bdl	34.1±18.1	42.4±34.6	Bdl	172.9±148.7	51,100±400	997.4±102.1	Bdl
C37	122.8±52.8	58.2±49.1	Bdl	45.7±20.1	505.5±9.8	41.2±3.5	19.5±8.4	Bdl	Bdl	Bdl	126.9±21.0	111.6±36.9	49.0±34.5	337.9±156.7	54,100±400	1200±100	128.7±87.4
C38	168.6±56.3	77.3±52.3	24.7±24.2	55.0±21.4	449.6±9.7	38.9±3.6	53.2±10.3	Bdl	Bdl	Bdl	91.3±21.6	69.6±39.2	Bdl	244.3±166.4	56,800±500	1800±100	Bdl
C39	163.9±56.0	108.5±52.1	Bdl	39.6±21.2	317.4±8.3	42.1±3.6	11.5±8.5	Bdl	Bdl	Bdl	147.6±22.7	115.1±39.8	Bdl	210.1±150.6	46,400±400	874.5±104.4	Bdl
C40	177.5±54.2	Bdl	26.8±23.2	60.7±20.6	404.1±9.0	37.0±3.4	10.7±8.2	Bdl	Bdl	Bdl	47.3±19.6	108.6±37.9	39.7±35.3	380.4±167.2	60,000±500	1200±100	233.6±94.6
C41	75.5±53.3	Bdl	Bdl	36.2±20.3	320.9±8.1	35.3±3.3	15.6±8.3	Bdl	8.5±6.3	Bdl	46.0±19.4	101.7±37.3	Bdl	507.3±171.1	62,900±500	1600±100	170.2±92.5
C42	150.6±52.9	75.2±49.3	Bdl	42.0±20.1	390.7±8.7	39.0±3.4	Bdl	Bdl	8.3±5.9	Bdl	64.0±19.6	97.7±36.7	74.8±35.7	220.9±174.8	69,300±500	1300±100	140.4±91.8
C43*U	Bdl	Bdl	Bdl	Bdl	492.7±10.6	44.4±4.9	19.7K±100	Bdl	Bdl	Bdl	172.2±25.0	295.7±44.1	Bdl	356.6±165.6	52,600±500	677.2±110.5	Bdl
C44	345.9±83.4	211.2±77.2	96.7±35.8	70.5±31.5	600.2±14.3	17.4±4.1	16.6±13.1	Bdl	Bdl	Bdl	Bdl	126.3±64.9	Bdl	334.2±183.8	39,300±500	502.3±128.8	Bdl
C45	137.8±57.8	Bdl	Bdl	29.3±21.9	284.9±8.1	40.0±3.7	Bdl	Bdl	Bdl	Bdl	Bdl	124.4±41.6	Bdl	318.2±193.3	74,500±500	1400±100	114.4±100.4
C46	81.0±65.3	Bdl	Bdl	27.0±24.9	405.2±10.2	23.7±3.5	Bdl	Bdl	Bdl	Bdl	65.4±24.9	103.4±49.3	Bdl	329.6±124.6	23,200±300	501.0±99.4	Bdl
C47	281.8±75.9	194.1±70.4	92.9±32.7	46.1±28.6	621.6±13.7	36.7±4.5	Bdl	Bdl	Bdl	Bdl	57.4±29	86.8±57.3	Bdl	Bdl	46,000±500	710.0±128.7	Bdl

All values are in parts per million (ppm). Bdl = below detection limit. ± Values = the detection limit.

* U = Unglazed surface, interior of the ceramic pot. This analysis is still affected by the exterior glaze.

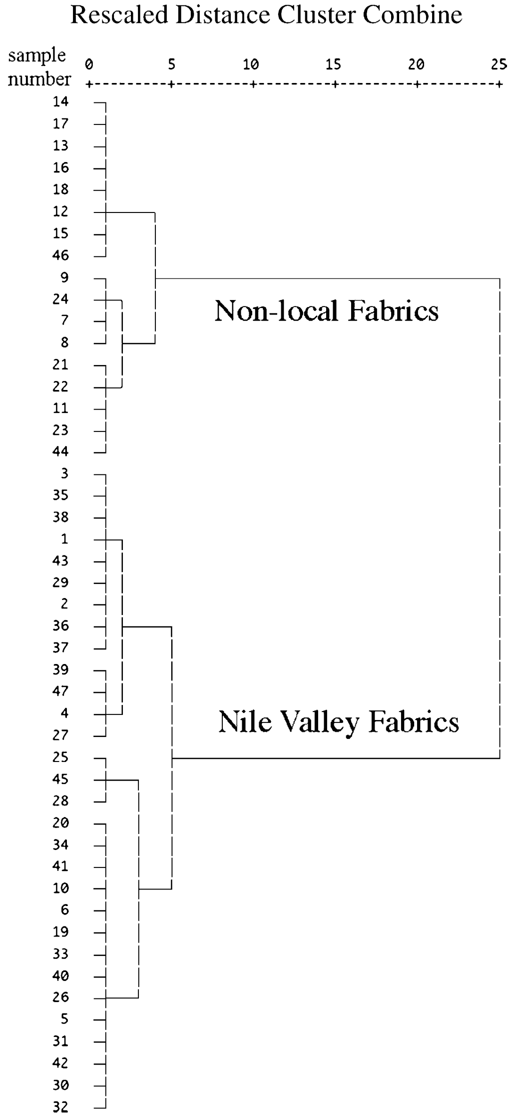

Fig. 3. Dendrogram using average linkage (between groups), squared euclidean distance for the following 11 elements: Sb, Sn, Ag, Sr, Rb, Pb, Zn, Cu, Co, Fe, and Mn.

wares that are not Nile Valley wares. Ultimately we should be able to be more specific in our classification of results. First, however, we need to build a broader library of the geochemical attributes of an appropriate sampling of wares from other archaeological sites in the Nile Valley, including the Nile Delta. It is also important that this geochemical library be complied using similar geochemical tools (a portable EDXRF unit). As the presently available geochemical sampling universe is quite small, confined to El Hibeh itself, we cannot yet classify specific Nile Valley sources, such as El Hibeh.

It is well established from prior research that, in terms of compositional analysis, Egyptian ceramics may be divided generally into three major groups: 1) marl clays; 2) Nile silts (dominantly derived from igneous rock detritus); and 3) "other," a grouping sometimes dominated by imported pottery (whose clay sources often derive from marine clays, desert environments or volcanic terrains), but which also may include "mixed" Nile silt and marl clay fabrics, wadi clays, and other clay sources (see, inter alia, Refs. [1–5,8]). Knowing this, we focused our investigation on the results for three of the eleven useful elements obtained from the NITON unit analysis: rubidium (Rb), iron (Fe) and strontium (Sr). All three had very good signal-to-background parameters. More importantly, all three had anticipated relationships to the geochemical sedimentology of Egypt, as well as the local geochemical sedimentology of the El Hibeh region, that we expected to see reflected in the three basic ceramic types noted above. Specifically, strontium embodied information about carbonate content, rubidium about volcanics and illitic mud, and iron about non-carbonate silt detritus (see Section 4).

The geochemical data for rubidium, iron and strontium were studied by use of a triangular scattergram, produced in Delta Graph 5 (Fig. 4). The triangular scattergram was set up so that one corner, using Sr, demonstrates carbonate content; one corner, using Rb, indicates volcanics and illitic mud; and one corner, using Fe, reflects non-carbonate silt detritus (dominantly derived from igneous rocks). In order to plot all three elements on the same graph at a reasonable scale, they were modified as follows: no change for Sr (ppm); Rb (ppm) was multiplied by 10; Fe (ppm) was divided by 100.

4. Results and discussion

The results of the petrographic data collection are presented in Table 1. Table 2 shows the results of the geochemical analysis. Both the petrographic characteristics and the chemical composition of the ceramics are a function of their source materials, which originate from various combinations of natural sediments, crushed rock, and organics. Geochemically, each source component has its own particular chemical signature, which is a function of the unique history of that substance, a history that includes near surface processes such as erosion, transport, and deposition. For ceramic studies, it is important to be able to distinguish among the petrographic characteristics and the chemical signatures of the individual different natural sediments and rocks that are incorporated in the ceramic end-product. For Egypt, this generally means identifying marl clays, Nile silts, desert soils, volcanic tempers, igneous rock tempers, marine mudstones, sandstones, and evaporites.

In addition, it is important to note that the chemical signatures of the different natural sediments also reflect the geological environments of the ceramic source materials, potentially to a very closely defined environment. Put another way, we should be able to reconstruct

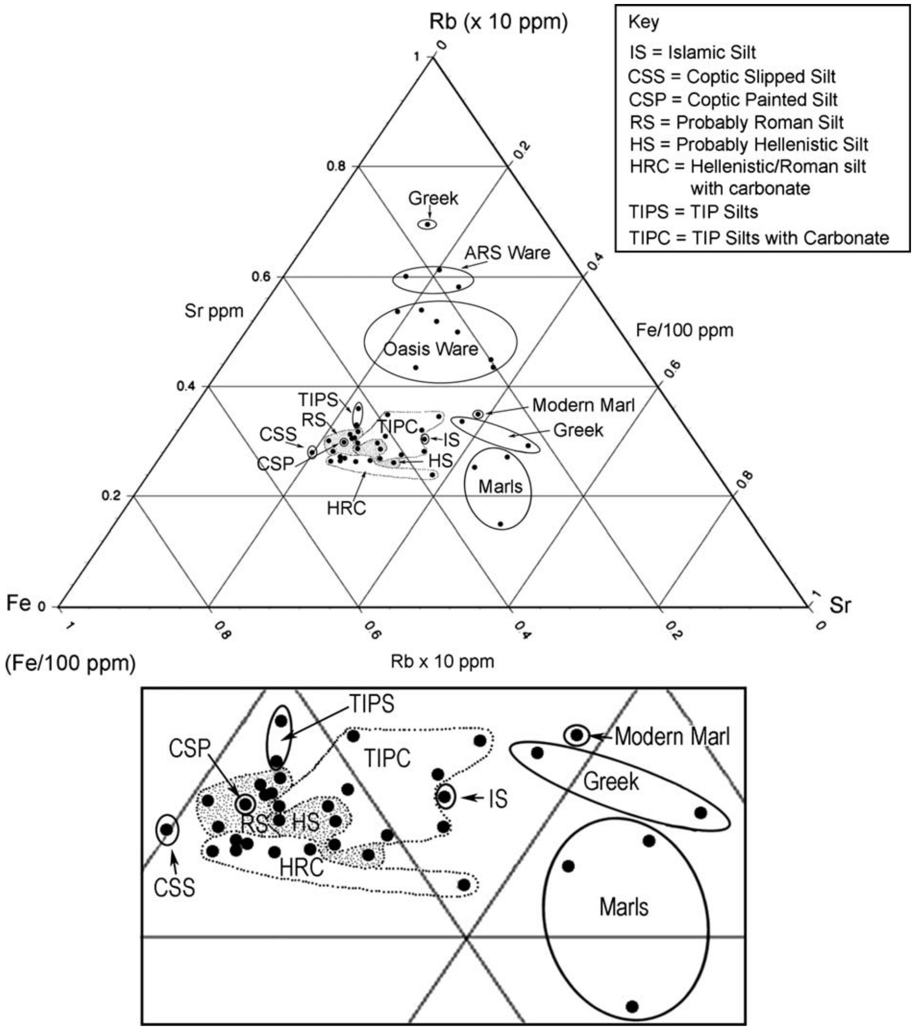

Fig. 4. Rb-Fe-Sr scattergram showing the classification of pottery by portable NITON Xlt-793W EDXRF analysis.

the geological environment(s) of the ceramic source materials from their geochemical signals. It is for this reason, explained in greater detail below, that we chose to concentrate on Rb, Fe and Sr in our geochemical analysis of the ceramics in this study.

5. Geochemical sedimentology at El Hibeh

The site of El Hibeh and its environs are a mixing zone for two petrologic sedimentary provinces or facies: 1) the Nile flood plain; and 2) desert limestone/marl terraces and platforms. The characteristics of these two petrologic provinces in the Hibeh region are summarized below.

The Nile River sedimentary facies is divided into two subfacies: 1) a coarse-grained quartz and quartz polymorph gravel subfacies common to high-energy Nile channel environments; and 2) a fine-grained subfacies that accumulates as overbank sedimentation and in stagnant Nile backwater environments. The second, low energy subfacies is dominated by relatively well-sorted, fine-grained clastic alluvium that consists of quartz with minor amounts of feldspar, quartz polymorphs and heavy minerals (pyroxenes, amphiboles, and magnetite-ilmenite). The energy of the local Nile River depositional environment controls the deposition of the clay fraction; clay and fine silts will settle only in the low energy environments. The parent sources of the minerals present in Nile silts are the granitic to basaltic igneous rocks associated with the White and Blue Nile highlands, plus bank erosion along the long transport path, plus local inputs of wadi sedimentation. In northern Middle Egypt, local wadi sedimentation input is confined largely to desert facies sedimentary materials, which are dominated by clastic limestone debris.

The second sedimentary province in the El Hibeh area is the limestone/marl terraces and platforms of the desert. The great bulk of the desert sediments in the Hibeh area were formed by mechanical disintegration (from wind, overland runoff, and exfoliation due to solar exposure) of the limestone and marl beds that are exposed in vertical cliffs in the region. These cliffs were cut both by the Nile River and by the water flow that created the perpendicular-trending dry wadis that flow into the Nile River basin from the Eastern Desert. Chemical weathering in the area, in contrast, is minimal as the region is dominated by aridity. Despite this dominating aridity, however, there is ample evidence that generation of minor soil moisture does occur in the desert. This evidence includes the appearance of spatial-cycling selenite deposits in the desert soils, C-horizon development on stable desert terraces, and rain etch marks on alluvial limestone pebbles and cobbles found in armored desert pavements.

The limestone outcrops at El Hibeh belong to the Qarara Formation limestone facies of the Mokattam Group (lower upper Eocene to upper middle Eocene). This local material is a massive foraminiferal packstone-limestone, with interbedded wackestone-limestone-marls [6,7,9,10]. "Packstone" refers to a limestone with a self-supporting granular framework; "wackestone" refers to a very poorly sorted, lithified biocarbonate hash with a significant clay matrix content; and "marl" is a loosely applied term for an unspecified and unconsolidated mixture of clay and calcium carbonate.

The color of the local El Hibeh packstone varies from grayish-white to brownish-yellow. Petrographic evidence indicates that the browning coloration of the packstone is a function of goethite (iron oxyhydroxide) mineralization, which is spatially associated with the mud-fraction facies within the limestone. Petrographic evidence also indicates that the greater the clay content, the greater the iron oxyhydroxide (goethite) concentration and the browner the color of some of the marl horizons that occur between the limestone beds. There are also, however, marl horizons between limestone beds that are light to dark gray in color, and show no to very little evidence of iron mineralization. In addition, there are hydrothermally altered marl clay facies in dike swarms that are mineralized by significant concentrations of hematite and goethite. These deposits are iron enriched and are found in the local desert within the boundaries of the El Hibeh site. Consequently, iron concentrations vary considerably both in the packstones and in their interbedded clay horizons.

The desert soils at El Hibeh are dominated by a pebble to cobble-sized coarse chert fraction that is commonly coated by desert varnish on the stable upper exposed surface of the clastic and by a hematite stain on the underside. Occasional granule to pebble/gravel-sized polished quartz occurs in the older Pleistocene Nile terraces, but fine-grained Nile River sand and silt clastics are not generally observable, as they have been winnowed out of the system. The bulk of the soils below the mantled surfaces are poorly to very poorly-sorted limestone cobbles and sandy silts, diagenetic carbonates, marl clays, and nodular and bedded chert fragments and pebbles. Within the anthrosols associated with cultural activities at the site, there is the addition of organic detritus, bone, pottery debris and igneous rock fragments dominated by granites, granodiorites, diorites, monzonites, and metamorphic greenstones and quartzites. These anthrosols spill over into the desert from the boundaries of the El Hibeh tell mound and from desert burial activities. In general, though, the desert system is characterized by a clastic limestone component in a silty clay matrix, which together form a mechanical admixture of the limestone and marl beds from which the sediments are derived.

6. Geochemical sedimentology and rubidium, iron and strontium geochemistry

Three elements, rubidium (Rb), iron (Fe) and strontium (Sr), were chosen from our geochemical analysis as broadly representative of the geochemical sedimentology at El Hibeh (discussed above)—as well as the Nile Valley in general—or the sedimentary ceramic fabrics collected at the site or both. Iron is generally loaded in the Nile silts (Nile River sedimentary facies) and also occurs in some of the imported fabrics. Strontium is highest in limestone and marl sediments (desert facies). Rubidium dominates in the imported fabrics containing marine mud, volcanic rocks and minerals and evaporates, and also occurs to a lesser extent in the Nile silts. By constructing a triangular diagram using these three elements, therefore, it proved possible to separate out groups of ceramics by origin, by fabric type, by date, and sometimes even by form (Fig. 4 and below). Reasons for the environmental correlations with these three cations are discussed below.

Rubidium is loaded in fabrics of our sample set identified as Greek imports, African Red Slip (ARS) ware, and Oasis ware [3–5]. It is also loaded in the micaceous silt fraction of the Nile silt fabrics (Fig. 4). Understanding the geochemical behavior of rubidium is key to understanding why this is the case and why we chose rubidium as one of the key elements in our study.

Rubidium does not form its own minerals; rather, it generally acts as a trace element within other minerals, especially those minerals where it proxies for potassium. In addition, rubidium behaves similarly to cesium and is highly favored during ionic exchange reactions. It has a very low ionic potential (the ratio of ionic charge over the ionic radius ($Z/r = 0.68$)) and therefore tends to remain in solution during transport and sedimentation processes. Rubidium can proxy for potassium under a number of conditions that may be reflected in the geochemical fingerprinting of ceramics. For example, rubidium can proxy for potassium in illite clay, sericite (a fine-grained muscovite that forms from the weathering of the main primary igneous potassium minerals), potassium micas such as muscovite and phlogopite, leucite (a feldspathoid), and potassium feldspars such as sanidine (volcanic) and orthoclase and microcline (granitic). Montmorillonite is an expandable clay mineral that can acquire potassium and rubidium by adsorption (cation exchange). In addition, there are a variety of open-structure zeolites such as heulandite, phillipsite and clinoptilolite that can adsorb potassium and rubidium from solution. Potassium minerals such as sylvite and niter also form as evaporites. Niter principally forms in desert playa lake environments and is a constituent in many archaeological anthrosols associated with burial practices.

Rubidium concentrations are much higher for the ceramics identified as Greek imports because these are derived from geological terrains that contain volcanic minerals loaded with potassium minerals such as sanidine, alteration products of volcanic glass such as potassium and rubidium-adsorbed montmorillonite, and alteration products of potassium feldspar such as illite. In rare cases cation exchange zeolites are present. Oasis ware fabric samples, also with very high rubidium values, reflect the evaporitic conditions of the local source areas that have significantly elevated amounts of potassium mineral salts, in addition to illite and muscovite concentrations. Similarly, African Red Slip ware samples exhibit very high rubidium values because they likely derive from Tunisian illitic marine shale deposits.

Rubidium also is associated with fine-grained Nile sediments, especially the clay and mica mineral fractions of these sediments, and is loaded into the paste fraction of ceramics manufactured from Nile silts. Most of the Nile silt fraction in the silt sample sherds that were examined, however, consists of very fine-grained quartz, with only limited amounts of feldspar and feldspar alteration products. Consequently, the overall concentrations of rubidium are comparatively low, but still higher in value than is the case for sediments obtained from limestone weathering or for local marl clay fabrics, where kaolinite, which does not have potassium, is the dominant clay mineral. Additionally, it is important to note that Nile silt rubidium values are sensitive to minor fluctuations in annual Nile flood sedimentation and consequently have the additional value of potentially being able to distinguishing between different Nile silt deposits.

Iron in Nile silts is derived from the heavy minerals (magnetite, titanomagnetite, pyroxenes and amphiboles) that are detrital components in the Nile silts, and from the weathering of those minerals in the transport and sedimentation processes. Iron also is present in intermediate and mafic volcanic rocks and in sediments tied in volcanic glass and minerals such as magnetite, iddingsite, goethite and hematite. Iron has two ionic charge states, $+2$ and $+3$, with ionic potentials ranging from 2.70 ($+2$ state) to 4.69 ($+3$ state). The divalent iron remains in solution during normal transport and sedimentation reactions, but the trivalent state commonly hydrolyzes to form oxyhydroxides. In natural Nile silt sediments, iron is present as an oxidized/hydrated mineral phase, generally as a component of the clay-size fraction (goethite, hematite, and maghemite). Iron also occurs in heavy detrital minerals such as magnetite that are in the fine fraction of Nile alluvium and in sediments derived from volcanics. In limestone/packstone iron is present as a pseudomorph after pyrite. Iron concentrations in carbonate sediments (packstone and marls), however, are far lower than in Nile silts (with the exception of the hydrothermal deposits of the desert facies).

Nile silt fabrics contain the highest iron concentrations in the tested El Hibeh ceramics. Iron therefore was chosen as one of the key elements of our analysis because of its ability to distinguish Nile River sedimentary facies (and potentially the local El Hibeh Nile River sedimentary facies) from the desert carbonate facies (and potentially the local El Hibeh desert carbonate facies). Some imported or non-local fabrics also might have high iron concentrations resulting from weathering phases of volcanics, but none were among the samples that we have studied here. Oasis wares, Greek imports, and ARS wares, however, tend not to be overly enriched in iron.

Strontium is loaded into the carbonate desert facies at El Hibeh and elsewhere but occurs only in fairly low concentrations in the Nile silts. Strontium has an ionic potential ($Z/r = 1.79$) that is between rubidium and divalent iron and therefore tends to remain in solution during transport and sedimentation. It is geochemically associated with calcium and acts as a proxy for calcium in most reactions. It is therefore abundant in calcium carbonate derived sediments such as the El Hibeh desert sedimentary facies, detrital limestone/packstone, reef limestone, soil caliche deposits, and micritic clay deposits (marls). One of the primary minerals formed by strontium is strontianite (strontium carbonate), which develops in hydrothermal deposits and as concretions in limestone and marls. In addition to strontianite, strontium also forms the sulfate mineral celestite (strontium sulfate), which occurs in evaporite deposits, limestone and hydrothermal deposits. Additionally, strontium forms in desert lakebeds with gypsum and halite. In summary, strontium tends to occur with sedimentary carbonate facies, marls, and some evaporites.

The sedimentary behavior of strontium is the inverse of iron, so together these two elements act as very effective classifiers to separate marl sources from Nile silts. One of the more interesting aspects of marls is that they contain differing quantities of carbonate and sulfate, and thus different deposits show significant variance in strontium contents. It appears, however, that the range in strontium concentrations in single sources is rather narrow, which favors the ability of strontium to serve as a chemical fingerprint for determining the provenance of marl ceramic fabrics.

7. Ceramic fabric classification with rubidium, iron and strontium geochemistry

Using the triangular scattergram with Rb, Fe and Sr (Fig. 4), it is possible to discriminate a series of geochemical ceramic groupings or potential groupings (some had only one sample) among our sample set. The ARS ware, the imported fabrics from the Aegean and the Oases ware separate out very clearly, as do the marls. The remaining main grouping represents silt fabrics with and without carbonates (Coptic sherds, Third Intermediate Period silts with and without carbonates, and Hellenistic and Roman silts and silts with carbonates). It is highly likely that at least some, and possibly all, of the samples belonging to this main ceramic grouping were manufactured at Hibeh from local clays. Certainly the chemical signatures are well grouped with respect to our three-element scattergram spatial distributions (Fig. 4) and are clustered (11-element hierarchical cluster analysis, Fig. 3) to suggest that the sedimentary sources may be very local and at the very least lie within the confines of the Nile river regime (Nile Valley and Delta). As previously stated, further investigation is necessary, however, before we can attribute, with any degree of security, all of these ceramics to local production at El Hibeh itself. Additional geochemical and petrologic field data must be collected, not only from El Hibeh but also from other sites in the Nile Valley and Delta. Furthermore, the implications of the data and findings for ceramic production and distribution patterns through time and space also must be considered in greater detail.

It is possible to distinguish temporal differences within the main grouping of silt fabrics in our sample set (Fig. 4). In particular, one may easily differentiate Third Intermediate Period (TIP) and Hellenistic/Roman Nile silts. The TIP fabrics tend to have greater concentrations of rubidium than the Hellenistic/Roman ceramics. This is presumably a function of greater concentrations of illitic clay/muscovite mica (where the rubidium would be concentrated) in the TIP source area.

Fig. 5 summarizes the general geochemical separation of Rb, Fe and Sr based on the physiochemical and geological factors previously discussed. Ceramic fabrics that are derived from Nile flood plain and desert sedimentary facies systems are classified using iron and strontium, as discussed above, to distinguish Nile silt from marl fabrics. Both of these also may be distinguished from combination fabrics—"mixed" fabrics that combine Nile silt and marl clay and Nile silt fabrics to which calcium carbonates have been added. Such combination fabrics might be natural (Nile silts with desert soil debris that has washed into the Nile lowlands) or man-made (the potter mixed Nile silt and marl clays, or the potter tempered Nile clay with carbonate clastics). The combination of rubidium (higher in the pure silts) and strontium (higher in the carbonate facies) clearly distinguishes this group.

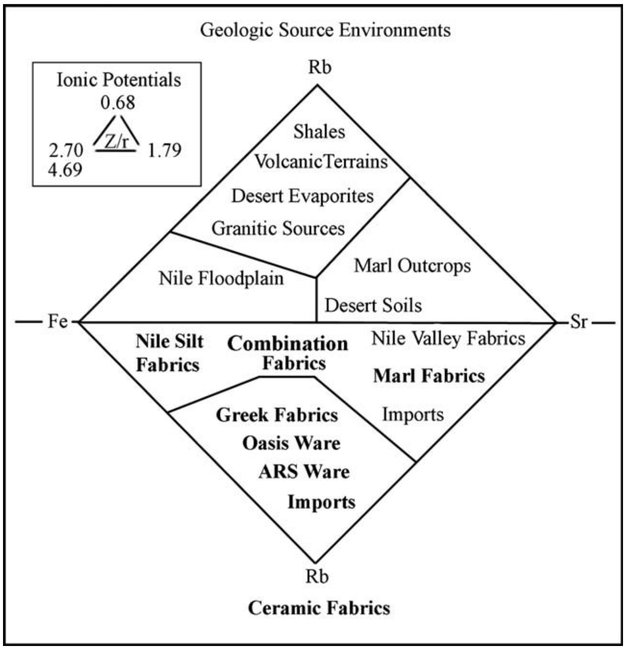

Fig. 5. Geochemical separation of Rb, Fe and Sr based on physiochemical factors.

8. Summary and conclusions

The Rb-Fe-Sr geochemical signature proved able to discriminate among our ceramic sample set to a surprisingly sensitive degree. Our triangular scattergram based on these three elements provides excellent temporal and spatial separation for fabric type, origin and date. This is the case because rubidium, strontium and iron reflect key sedimentary characteristics not only of the ceramics themselves but also of the geochemical sedimentology from which the ceramics derived.

Rubidium provides an important elemental signature because of its geochemical behavior as a proxy for potassium in detrital sedimentary minerals such as potassium-feldspars, illite, muscovite, and evaporites. This cation provides for the characterization of volcanic, evaporitic, illitic mudstone/shale, and granitic terrains that provide source detritus to the clay/paste and sand/temper deposits exploited by potters. Iron is more often highly loaded in the silt and clay fractions of Nile silts as well as other non-limestone sedimentary detrital/in-situ lag clay deposits. Strontium, on the other hand, proxies for calcium in limestone and marl deposits and therefore acts as a classifier for marl and combination fabrics. Generally, the greater the concentration of calcium carbonate (calcite and aragonite), the greater the strontium signal. Together, the iron and strontium cations provide reasonably good separations between the carbonate and non-carbonate sourced sediments.

It is now time to return to our original three questions that generated this test study. The first two questions asked whether a meaningful geochemical signature could be obtained from Egyptian ceramics that, with the aid of optical petrography, would be discriminatory for provenance, time period, and clay type, and whether this signature could be obtained using a portable system suitable for field use. The answer to these two questions is a resounding yes.

Acquiring geochemical signatures of ceramics in the field using the portable EDXRF (NITON XLt-793W) spectrometer is not only a viable method, but also one that is non-destructive and that produces rapid and compelling results. The portable EDXRF, in fact, produced far better results than we had expected, and for Egyptian ceramic studies we have found that the NITON XLt-793W provides the most acceptable method of data collection.

In countries where radioactive sources are acceptable, the NITON 300 series single and multiple isotope source units provides an alternative list of elements and excellent detection limits. Laboratory-based EDXRF, ICP-MS, and INAA units provide the most ideal world of data collection, but only when it is permissible to transported samples off the archaeological site.

The answer to the third question, can the chemical signatures identify Nile Valley ceramics, is also yes. With further investigation and comparative data from a large universe of archaeological sites we hope to be able to fine-tune our analysis further and establish whether our main grouping of silt and combination silt fabrics was produced at a specific site, such as El Hibeh, or not. With respect to local El Hibeh ceramic production, all that can be said for sure at this point is that the samples are well grouped and closely clustered, and that the analytical techniques we are using, by relating the physicochemical, geochemical and geologic characteristics of end product and source material, show great potential for very close sourcing.

The next steps in our research are to continue to develop a geochemical profile of the El Hibeh ceramics, to enhance our understanding of the local geochemical sedimentology at El Hibeh and to refine the ceramic typology for the site. In addition, we are working on testing the broader usefulness of our analytical techniques by examining material from other sites both within and outside the borders of Egypt and by developing a library of comparative data. In future, it may well prove possible in the field, after the pottery at a specific site has been well characterized, to classify and date previously non-diagnostic or minimally diagnostic pottery. The protocol for such an activity would need to be fully developed and tested, but it should be feasible for one person to analyze between seven and fifteen sherds per hour in the field using both petrography and portable chemistry procedures (including time for data recording). A significantly large data inventory thus may be acquired with a relatively short time investment.

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