Julia Carr-Trebelhorn
Ingels Pottery Materials Study
University of KY Department of Materials Engineering, with the support of the Kentucky Geological Survey, and the USDA Soil Survey
Professor Richard Eitel, University of KY Department of Ceramic and Materials Science
May 2013

Research Background and Goals:

The purpose of this study was to examine pottery materials found on a local site during a planned excavation. In the mid 1980s, a University of Kentucky sponsored archaeological dig took place on a Bourbon County farm with the permission of the owner, Mr. Donald Thompson, who also gave permission for the current site visit and offered insight into our work there. In addition, the fieldwork was carried out with the support and assistance of Nancy O'Malley, Assistant Director of the University of KY Museum of Anthropology, who led the original excavation. The goal of this research was to determine a better understanding of the nature of the pottery, the materials used, and to add insight into the working methodology of the Ingels pottery.

Historical Background:

The farm was established by James Ingels, who came to Kentucky at the start of the 19th century. Around 1820, Ingels built a two-story home on the farm with bricks made on the site. In addition to the other works of the farm, an investment was made in establishing a redware pottery on the farm near the household. The house and pottery were very likely to have been situated close to one another, and the source for the clay was also nearby. The redware pottery was one of the modes of currency for the farm, and was relatively ordinary, simple, and low-fired. The wares were fired on the farm site in a beehive kiln. The clay body material was likely milled on site before use, as a contemporary inventory recorded that a mill was purchased for the pottery. For both the bricks and the pottery, the source for the materials was located nearby, on the farm property. As a low-fired earthenware, it served basic needs, but was not refined, and remained porous. It's porosity and simplicity meant that it found use in daily activities, food storage, and food preparation, among other uses, but it was not expected to be particularly durable or to take a public role as a part of a dinner service. The pots were loaded onto wagons and transported throughout the central KY region, where they were likely exchanged for other goods, including old rags that were in turn traded for groceries, other goods, or possibly money at the paper mills. The rags were then made into paper that was sold or bartered. In the early 1820s, Kentucky was still remote, and had only limited access to the sorts of refined goods to which the Americans on the East Coast or the Europeans could easily purchase. By the 1840s, both trade and industry had developed enough that the redware pottery gave way to higher quality (and more vitrified) local ceramics as well as those from more distant places.

The farm sits on top of a fault line that runs approximately parallel to the current location of Bryan Station Road, and the soil in the area is classified as a Lowell Silt Loam, with a 6%-12% slope. This soil type is consistent over a large area in the county. A shallow, man-made basin was pointed out to us by the homeowner as the likely source of the clay body for both bricks and pottery. The man-made nature of this basin was confirmed by observations by Steve Jacobs of the KY Soil Survey during the site visits. Using a truck-mounted hydraulic drill provided by the State Soil Survey, soil samples were taken from four places. The soil samples were approximately 1"-2" in diameter, and extended down from the surface of the soil to the limestone bedrock. The soil depths are noted for each sample on Figure 2 (Soil Map), along with the GPS coordinates of each sample. They were all taken on the untouched perimeter of this basin and were used as outlined below for the series of tests.[1,2]

Methodology:

Analysis Types:

Several samples of pottery sherds were acquired from the original site, and are listed by site number in the Appendix. These sherds were tested using a variety of methods, but predominantly using X-Ray Diffraction (XRD) and X-Ray Fluorescence (XRF). Additionally, some powdered sherd samples were observed under DTA/TGA analysis to gain an understanding of the behavior and the firing history of the pottery samples, as well as to understand the behavior of the new soil samples in undergoing firing. A limited amount of SEM analysis was also undertaken, and the images are recorded in Appendix 3.

[1]However, the two samples nearest the house may have been affected to some degree by backfill from the relatively recent home construction that covers the site of the historic home previously on the same site. Care was taken throughout the study to acknowledge this potential factor, so Core Samples 3 & 4 are more frequently used than Core Samples 1 & 2.
[2]Additionally, the homeowner pointed out a potential location for a round beehive kiln, and initial explorations at the site suggested that this was a valid location to confirm by using other methodology. This GPS point is recorded in the appendix along with the four locations of the soil samples.

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The Soil Samples:

The soil samples were taken from a range of soil depths, extending down to the bedrock. Each sample was taken from a similar section of the material and from a central part of the larger core sample, in order to get a sample that was unaltered and approximately similar to other samples at the same soil depth. Illustration 1, below, shows the soil depths and the scheme for the sample numbers:

Illustration 1:

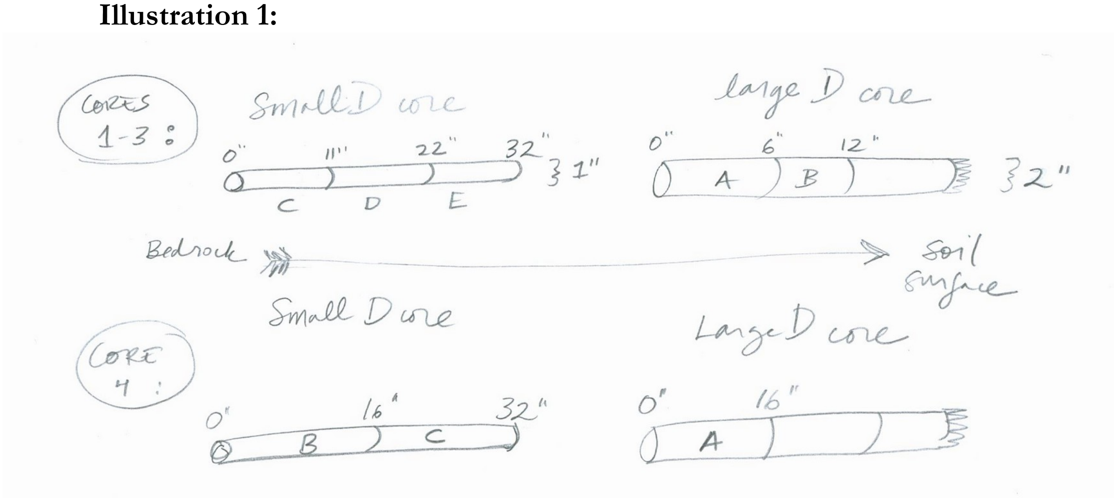

Table 3, below, shows the approximate soil depth of each sample gives the GPS coordinates for each core sample that was taken. These GPS coordinates are shown on the map in Appendix 2.

Table 3, Soil Sample Identification:

Sample Core Number	GPS Coordinates	Sample Name		Sample Region (in inches) from	Sample Region (in inches) from	
1	n38º07'10.6"	A		0	6	Large d core
1	W84º21'15.0"	B		6	12	Large d core
1	n38º07'10.6"	C		0	11	Small d core
1	W84º21'15.0"	D		11	22	Small d core
1		E		22	32	Small d core
2	N38º07'10.7"	A		0	7	Large d core
2	W84º21'14.6"	B		7	14	Large d core
2	N38º07'10.7"	C		0	11	Small d core
2	W84º21'14.6"	D		11	22	Small d core
2		E		22	32	Small d core
3	N38º07'9.4"	A		0	6	Large d core
3	W84º21'14.4"	B		6	12	Large d core
3	N38º07'9.4"	C		0	8	Small d core
3	W84º21'14.4"	D		8	16	Small d core
3		E		16	24	Small d core
4	N38º07'8.8"	A		16	24	Large d core
4	W84º21'15.1"	B		0	12	Small d core
4		C		12	24	Small d core

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X-Ray Fluorescence - XRF:

X-Ray Fluorescence was used to determine a chemical composition for the sherds. Following a series of XRD tests, the same powdered soil and sherd samples were pressed into a series of round pellets surrounded by a layer of solid boric acid, which were then inserted into the XRF holders and tested in one series. [3]

The following data (Table 1) was recorded using XRF at the Kentucky Geological Survey Laboratory, University of KY:

Table 1: Ingels Pottery & Soil Samples X-Ray Florescence, Recorded Data

XRF Data Ingels Pottery (Weighted)											
Sample	Type	SiO2 (%)	Al2O3 (%)	Fe2O3 (%)	Na2O (%)	MgO (%)	P2O5 (%)	K2O (%)	CaO (%)	TiO2 (%)	MnO (%)
BBC1AX	soil sample	71.58	14.02	9.09	0.29	0.77	0.22	1.77	0.33	1.93	0.10
BBC1BX	soil sample	72.66	14.73	7.31	0.41	0.93	0.17	1.85	0.39	1.57	0.08
BBC1CX	soil sample	60.48	19.40	9.48	0.34	1.73	1.54	4.32	1.46	1.26	0.12
BBC1DX	soil sample	70.21	16.45	6.82	0.30	1.08	0.27	2.46	0.64	1.76	0.27
BBC1EX	soil sample	65.66	17.39	10.97	0.29	0.96	0.23	2.33	0.40	1.76	0.00
BBC2AX	soil sample	70.98	15.90	7.65	0.28	0.87	0.22	1.86	0.33	1.91	0.02
BBC2CX	soil sample	57.64	21.25	9.41	0.41	2.63	0.49	5.92	1.06	1.20	0.18
BBC2DX	soil sample	53.15	20.42	10.94	0.34	1.92	5.48	4.36	2.35	1.05	0.00
BBC3AX	soil sample	67.00	16.65	10.50	0.31	1.01	0.19	2.32	0.34	1.69	0.14
BBC3CX	soil sample	58.77	19.91	9.71	0.33	1.65	2.74	3.79	1.91	1.19	0.11
BBC3DX	soil sample	56.24	20.19	12.30	0.37	2.07	1.04	5.22	1.43	1.14	0.09
BBC4AX	soil sample	66.22	17.33	10.01	0.32	1.08	0.16	2.85	0.38	1.65	0.04
BBC4BX	soil sample	62.38	18.82	11.50	0.32	1.33	0.28	3.41	0.57	1.40	0.08
BBC4CX	soil	67.05	20.02	4.49	0.36	1.63	0.26	3.68	1.06	1.45	0.03

[3]The soil and sherd samples were run in a single batch, using a pre-established program based on Shale standards, because no regional clay standard for testing has yet been established at the university. The Shale standards were developed to test mineral samples from the region, and were the closest developed methodology available with similar character to the clays and other mineral samples.

	sample										
15BB76 295	*pottery sherd*	72.90	14.26	5.64	0.41	0.95	1.03	2.35	1.05	1.40	0.20
15BB76 288	*pottery sherd*	68.35	10.95	9.90	0.41	0.30	2.69	3.54	1.85	2.11	0.32
15BB76 289	*pottery sherd*	62.29	12.52	13.80	0.32	0.40	3.07	3.65	1.79	2.20	0.34
15BB76 290	*pottery sherd*	66.35	11.99	10.13	0.29	0.37	3.26	3.70	1.79	2.12	0.22
15BB76 280	*pottery sherd*	58.32	11.89	14.25	0.29	0.32	5.59	4.44	2.91	2.04	0.62
15BB1025	*pottery sherd*	71.83	10.09	8.29	0.25	0.22	2.45	3.19	1.54	2.06	0.43
15BB102 3	*pottery sherd*	65.42	11.34	10.16	0.32	0.34	4.10	3.57	2.66	2.05	0.41

Table 2: Ingels Pottery XRF, Averages

XRF – Ingels Pottery Soil Samples – Mean:

SiO2 (%)	**Al2O3 (%)**	**Fe2O3 (%)**	**Na2O (%)**	**MgO (%)**	**P2O5 (%)**	**K2O (%)**	**CaO (%)**	**TiO2 (%)**	**MnO (%)**
64.29	18.03	9.30	0.33	1.40	0.95	3.29	0.90	1.50	0.09

XRF - Ingels Pottery Sherd Samples - Mean:

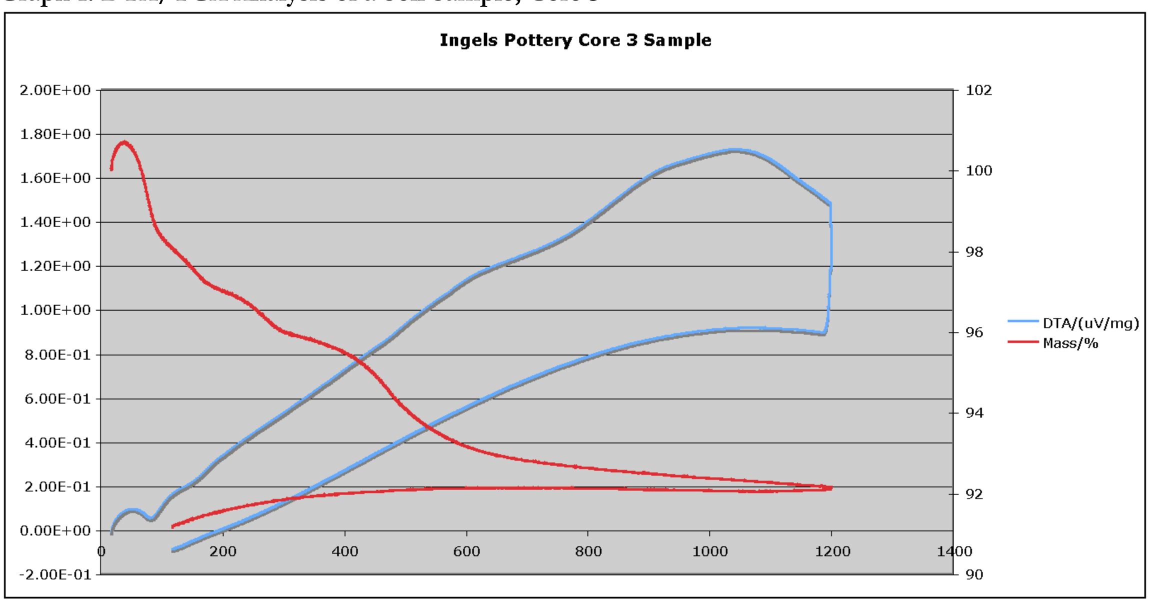
SiO2 (%)	**Al2O3 (%)**	**Fe2O3 (%)**	**Na2O (%)**	**MgO (%)**	**P2O5 (%)**	**K2O (%)**	**CaO (%)**	**TiO2 (%)**	**MnO (%)**
66.50	**11.86**	**10.31**	**0.33**	**0.41**	**3.17**	**3.49**	**1.94**	**2.00**	**0.36**

Although the contents vary by sample, in general, the silica content is approximately in the range of 60%-70%. Although the soil samples reached as much as 20% alumina, among the sherd samples the alumina percentages were typically lower, with the highest (15BB76-295) reaching 14%. In character, the sample 15BB76-295 is very similar to the 15BBC1A and 15BBC1B. The next most prevalent material is iron oxide, which was present in the sherds at up to 14% (15BB76-280), and among the soil samples at 12% (BBC3DX).

Differential Thermal Analysis/Thermogravimetric Analysis - DTA/TGA:

This testing was used both for the raw soil samples and on powdered sherd samples. The goal was to determine which phase transitions had taken place in the sherds, and to add insight into the firing temperatures achieved. The soil samples recorded data of the raw soil samples in order to determine when the clay body naturally underwent phase transformations, and at what temperature the clay body vitrified (See Graph 1, below.). Although the material did not produce the sharp peaks consistent with DTA/TGA analysis of pure clays, vitrification occurred between approximately 928ºC and at 1057ºC, and was complete by 1150ºC, before the end of the cycle at 1200ºC.

Graph 1: DTA/TGA Analysis of a Soil Sample, Core 3



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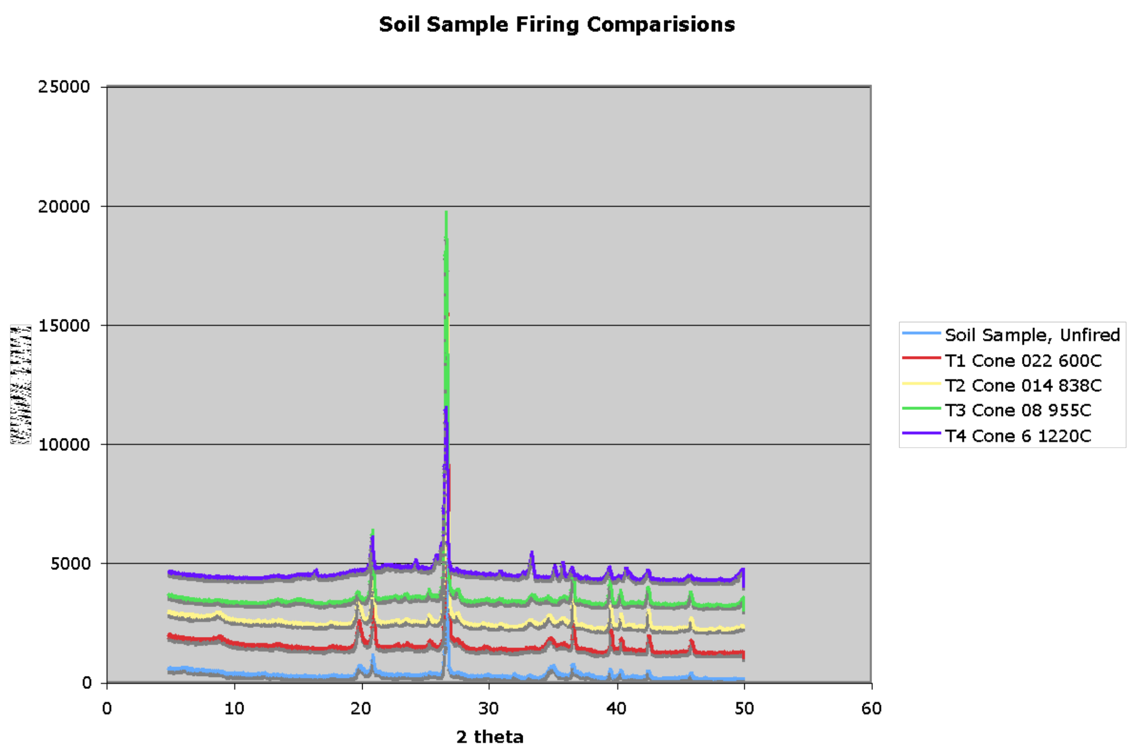
X-Ray Diffraction - XRD:

The predominant method of testing was XRD, which was used to establish an idea of the nature of the materials used, and also to establish a connection between the clay material located on the property and the pottery made on site. The pottery sherds all came from a series of locations at the same soil height of the excavation and all share similar characteristics, and none could be combined or reassembled into a reconstructed object. Some variations in color, softness, and fragility occur among the samples. All XRD testing was done on samples that had been ground by hand with a mortar and pestle into a fine powder using copper radiation. This was divided into two basic areas of testing, one for the original pottery sherds, and the second for the soil samples take from the site. Firing tests were made of the soil samples after mixing and milling in a ball mill in order to create a homogeneous clay slip. This slip was poured into small tile moulds and fired to four temperatures that define a possible working range for the clay body. These steps were undertaken in order to recreate some of the circumstances undergone by the original material and to observe the effects firing had on the structure of the clay body. The milled samples for Core 3 were cast into tiles, then fired to four temperatures that defined a potential range of firing temperatures for the pottery. Post-firing, these tiles were ground in the mortar using the same method as for the sherd samples. The series of XRD patterns below, in Graph 2, follows the material as it is fired to different temperature in order to observe phase changes. As phase changes occur, some peaks in the scan are diminished or disappear, and other small peaks emerge and sharpen as internal structures collapse or a new phase is created. By comparison between the sherd XRD scans and the soil firing temperature comparison chart (Graph 2), it is possible to determine a range of potential firing temperatures for each sherd. Temperature firing estimates for each sherd can be found in the Appendix.

Graph 2: Comparison: Unfired Soil Sample and Series of Fired Soil Samples, Core 3, Mixed and Milled



A complete set of XRD patterns for all sherds is located in the Appendix. A representative XRD pattern of a sherd (15BB76-289) with identifications of the most significant and likely mineral compounds is shown below, in Graph 3. Graph 4 shows a soil sample taken from sample from Core 3, Section B (from between 6"-12" of soil depth) that has also been analyzed. The XRD scans were analyzed using EVA software and mineral compounds were matched to the sample one at a time by looking at common materials in Kentucky geology as well as common clay minerals. Attempts were made to match as many minerals as possible to the scan, but the silica (quartz), being the most prominent material in the soil sample, has a dominating effect on the other minerals in the sample, and these become much more complex to determine with accuracy and certainty.

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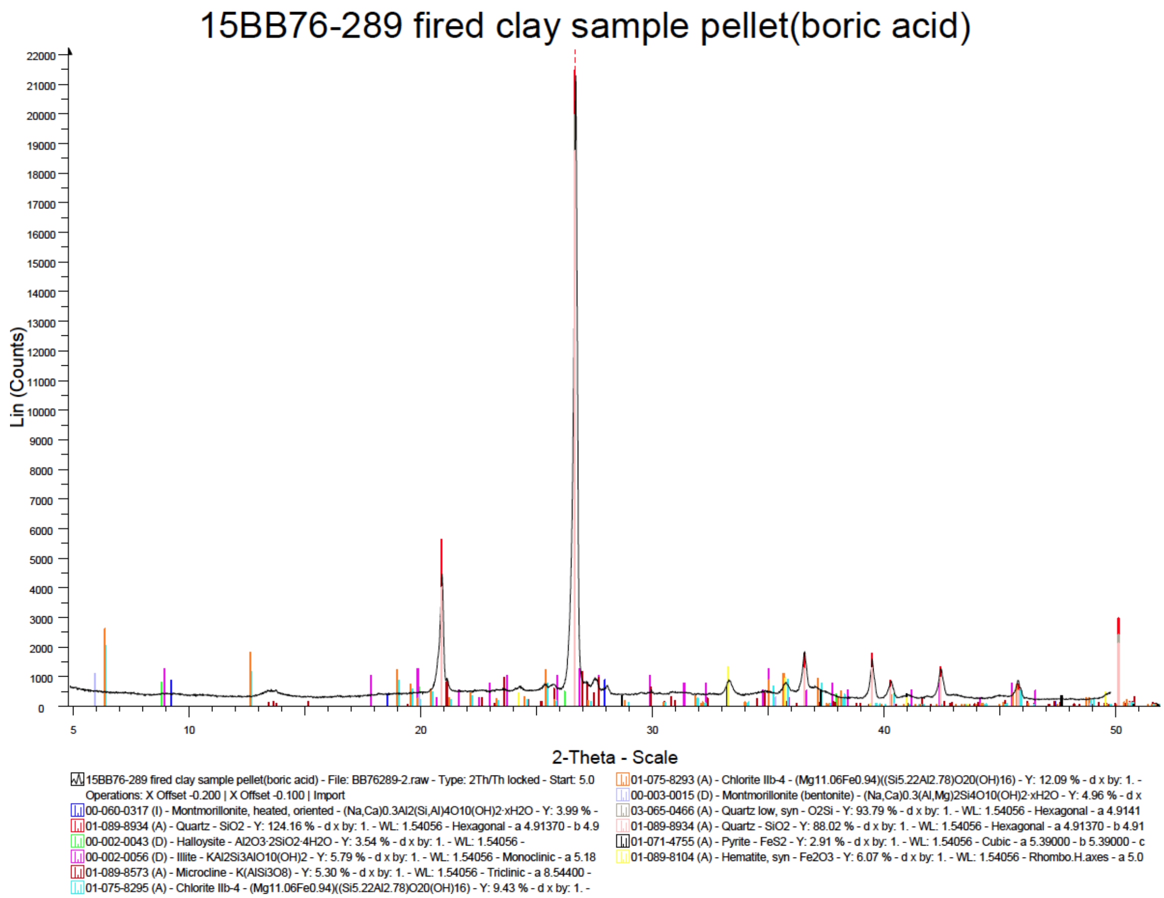
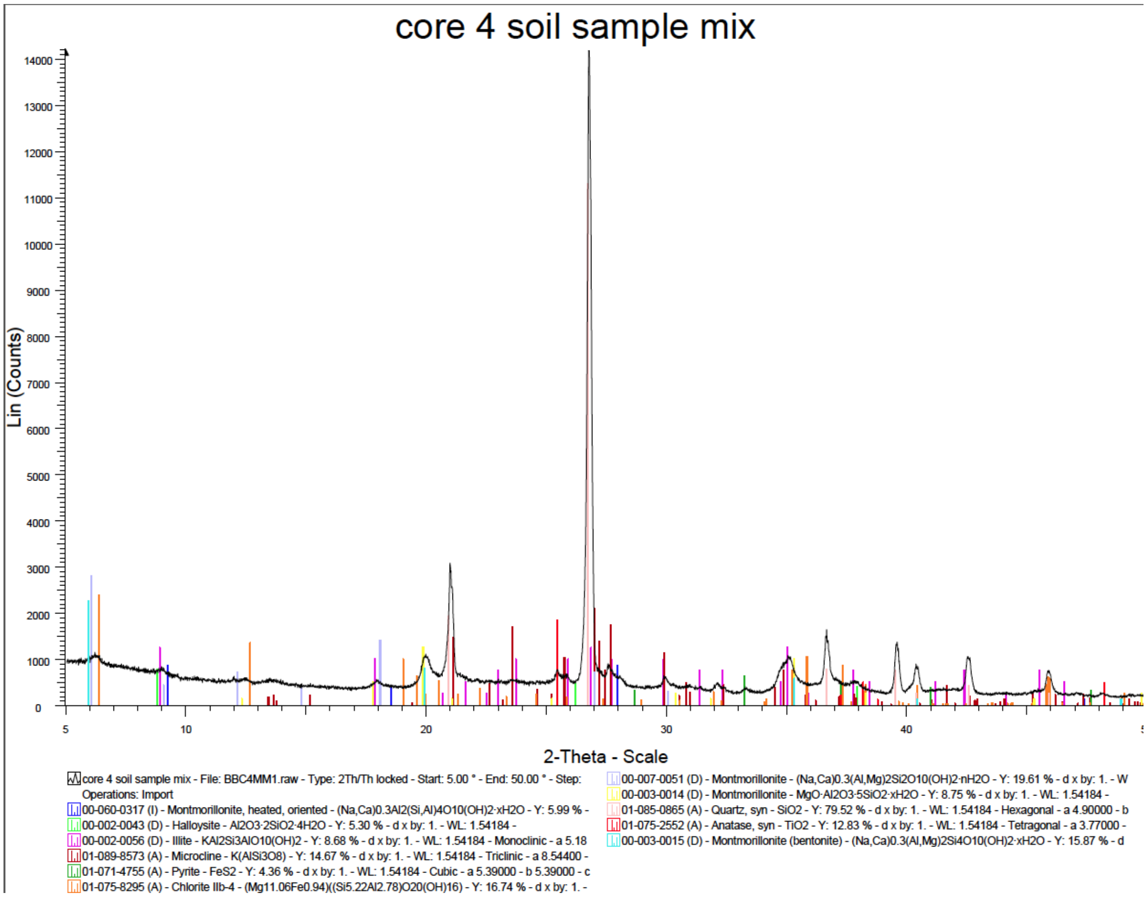
Graph 3: Sherd Sample with Mineral Identifications



Graph 4: Soil Sample with Mineral Identifications



Throughout the study, the XRD patterns were all approximately similar, particularly with respect to the largest peaks (specifically, quartz), although there were more variations among the intensities of some of the smaller peaks. The most significant series of peaks came from the quartz (silica), which comprises from 60%-70% of the samples, both from the sherds and the soil. Noticeably absent are significant peaks from kaolinite, and further analysis was done to determine if there were trace amounts not visible because of the intensity of the silica pattern peaks, or if the clay minerals took another form. The common clay-type minerals montmorillonite and illite are identified on the scan, along with halloysite. Iron is found as FeS (Pyrite) in the raw material sample, and appears in an oxidized form (hematite) in the fired sample. Chlorite, another potential source for iron, may be present, but it is difficult to conclusively identify, and peaks are often missing in XRD scans where it is known to be present. Another silica bearing mineral, microcline, is also identified. Because of the complex nature of the sample, and the difficulty in identifying the materials present in lesser amounts, not all peaks are definitively identified. The discussion of the clay fractions is completed below, along with Graphs 5-8.

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Using Separations to determine Clay Fractions:

A series of XRD scans were made on material that was isolated by a separation process in order to get a clay fraction distinct from the other soil material. Because the clay particles are significantly finer, a process that allowed the heavier particles to settle out of a solution was used to determine if kaolins were present among the more dominant compounds, such as quartz, that were present. This XRD series included a scan of (1) the unaltered clay fraction; (2) the clay fraction upon heating to 350 C; and (3) a glycolated clay fraction. The glycolation was used to re-establish any collapsed structures among the clay particles to see if processing methods such as grinding had altered any of the fragile clay structures, thus making the phases difficult to see. In these scans, the goal was to look for the peaks that would indicate a whether or not any of the finer clay particles could be seen in the soil material. These scans are shown in Graphs 5-8, below.

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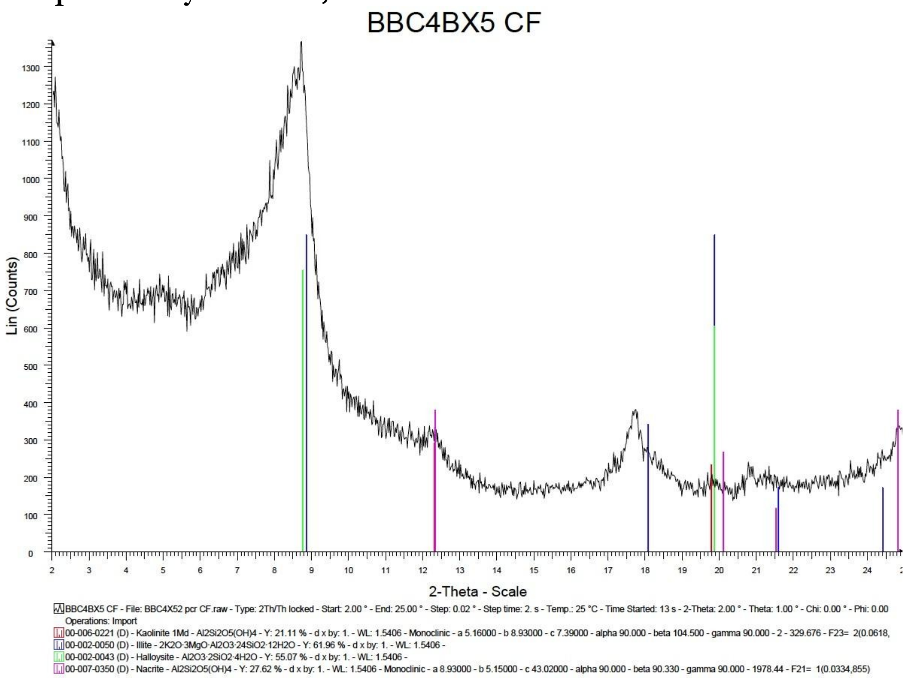
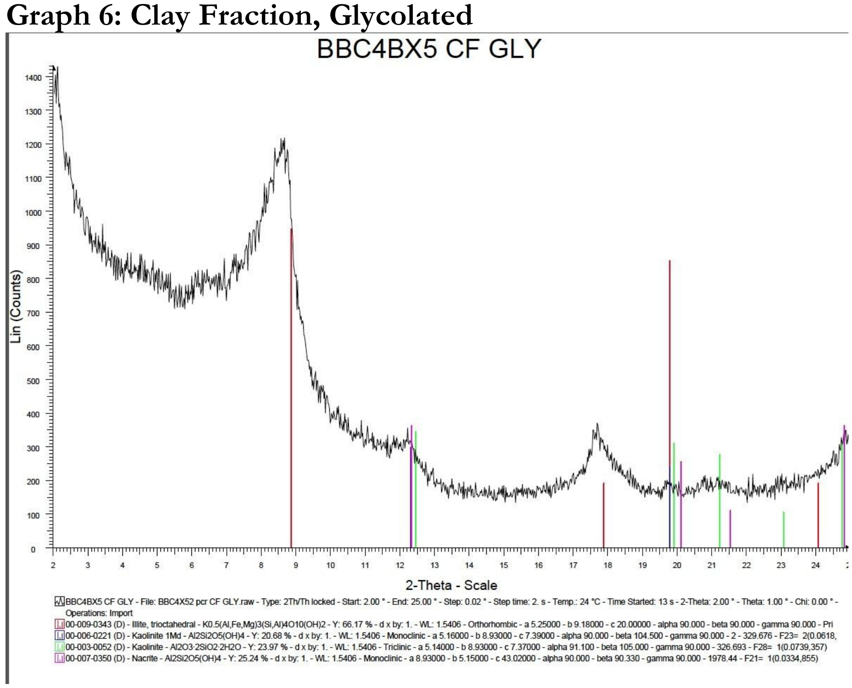
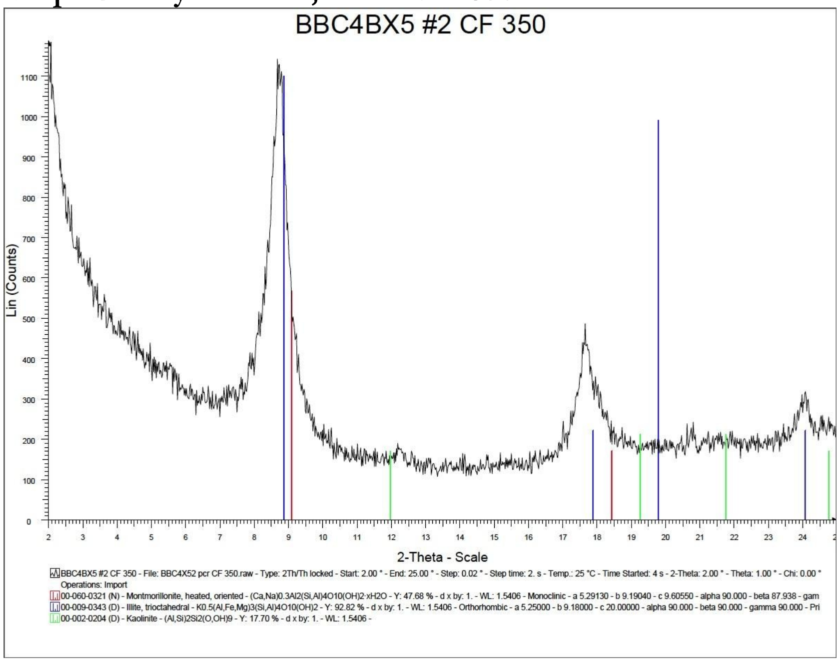
Graph 5: Clay Fraction, Unaltered



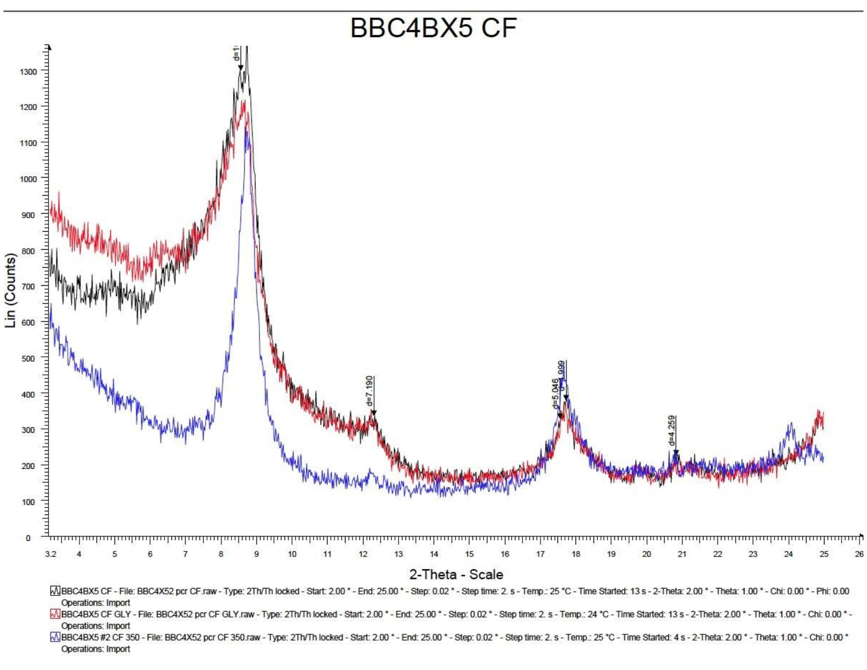
Graph 6: Clay Fraction, Glycolated



Graph 7: Clay Fraction, Heated to 350C



Graph 8: Clay Fractions Overlaid for Comparison



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The clay fractions indicate the potential presence of illite, halloysite, montmorillonite, and nacrite, but fail to show the presence of a substantial amount of kaolinite. It is important to note again that the mineral analysis of these scans is inconclusive based on the complex nature of the soil sample, but these clay-like minerals are known to the state of Kentucky and are common in soil samples throughout the region. As a clay body, in practice, this soil material is a semi-plastic substance that has a limited amount of workability, which suggests a less than "ideal" (kaolinitic) formulation, but it is well-suited to the making of basic earthenware in simple forms, such as those manufactured at the Ingels pottery.

<u>Conclusions:</u>

This research establishes a starting point for looking at Kentucky redware pottery and matches the soil samples from the site with the 19^{th} century sherds found at the site of the pottery. Additionally, it gives insight into the clay composition, such that, should a larger database exist, the pottery might be cross-compared with other regional pottery wares. However, with the information available at this point, it is not yet possible to attribute all redware pottery with a similar composition strictly to the Ingels workshop. Further testing on available and known samples to establish differences, as well as additional soil testing could be made to deepen the understanding and to begin to make more definitive analyses. By comparison between the fired samples and the sherds, as well as the DTA/TGA information, it is possible to establish a general firing range for the pottery as between approximately 900ºC to 1200ºC, which is an acceptable range for basic earthenware pottery as well as for the beehive kilns in use at the time.

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Appendix 1: Site Map (from Nancy O'Malley , Fall 2011)

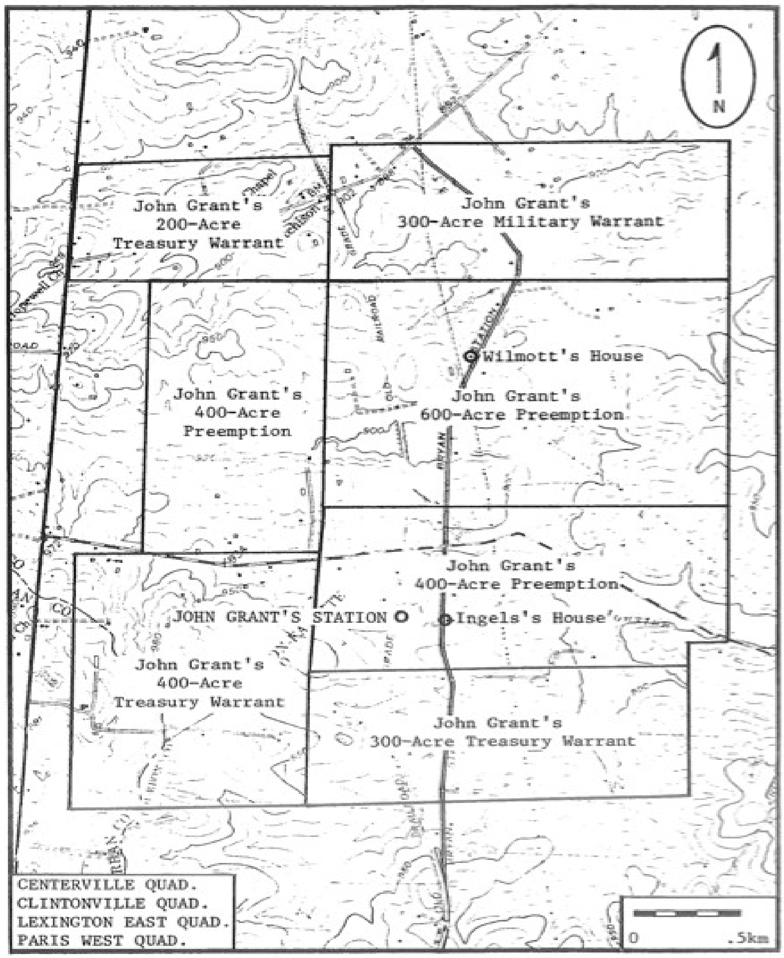

Figure IV-11. John Grant's station and land grants, Bourbon County.

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Appendix 2: Soil Map from Kentucky Soil Survey (Steve Jacobs, Fall 2011)

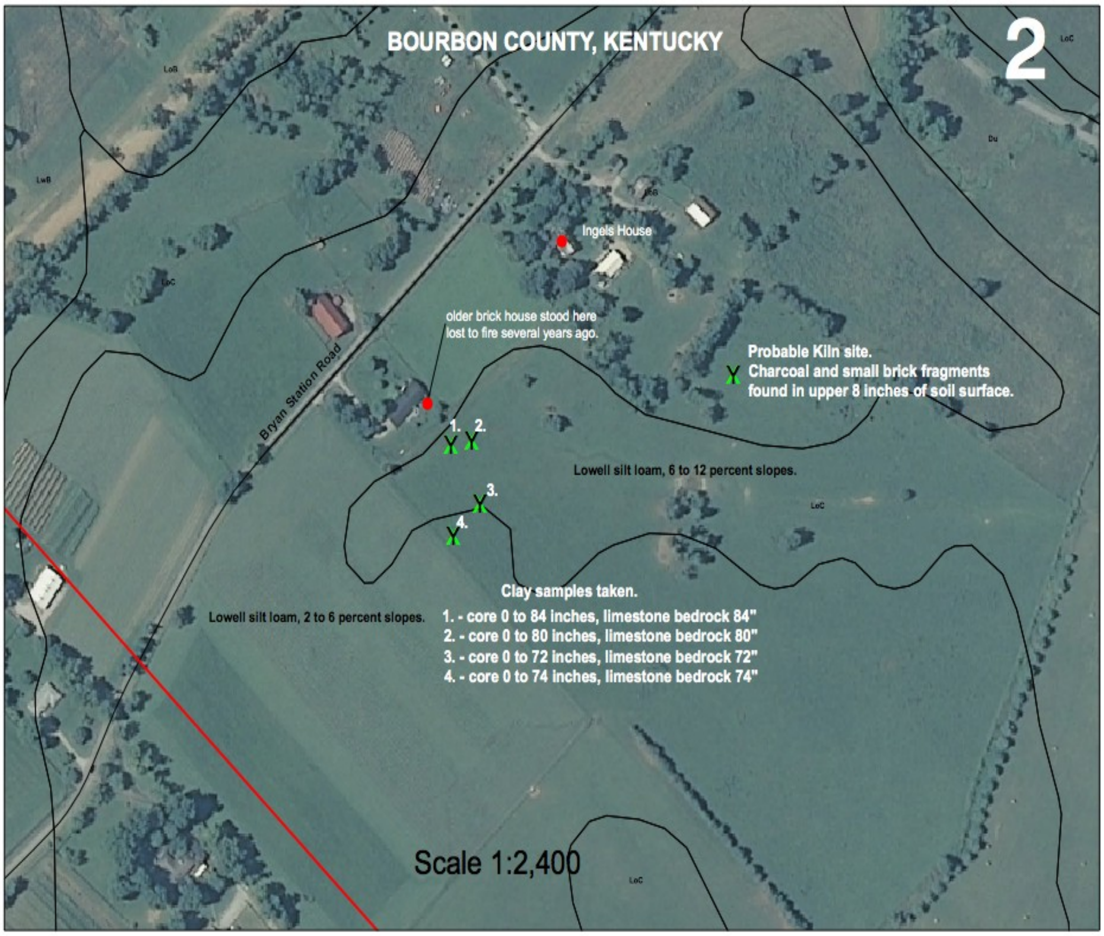

Figure 2: Location of the GPS Coordinates for Samples, Aerial View

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Appendix 3: Sherd Identifications

Sherd Sample Descriptions by Site Number

Sample Name	Description
15BB76/290	glazed both sides
15BB76/289	unglazed
15BB102/3	saggar base
15BB76/280	chunks of fired clay
15BB76/295	green glaze
15BB76/288	glazed one side
15BB102WPA	sherds from waster pit #1 glazed 2x redware
15BB102WPB	sherds from waster pit #2

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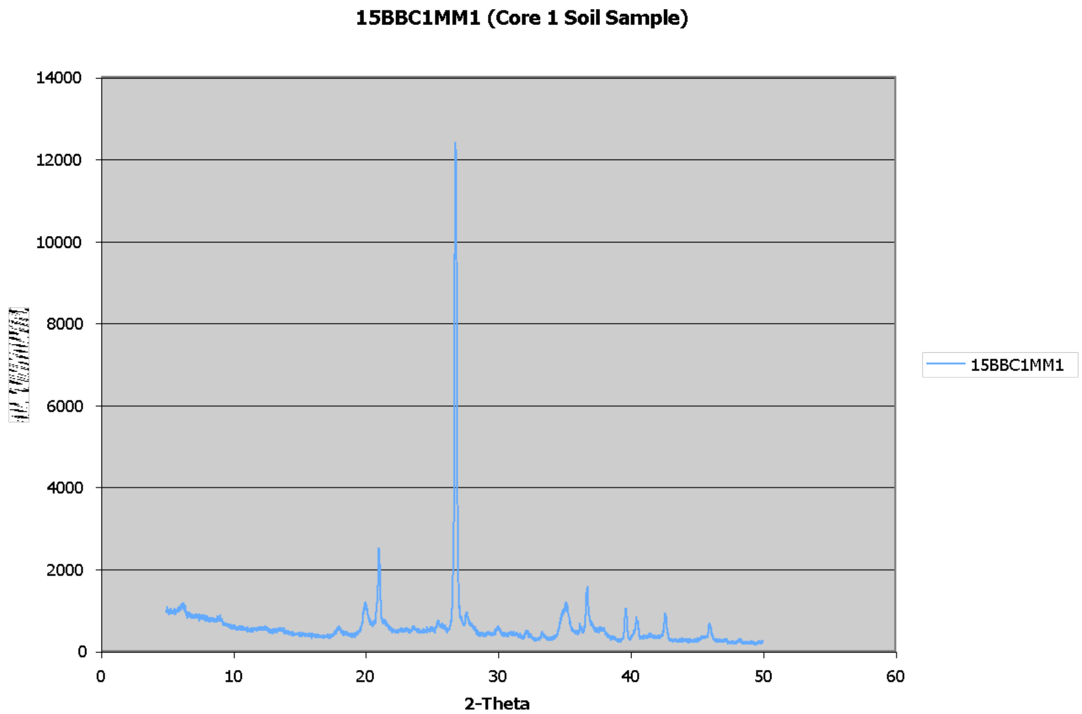
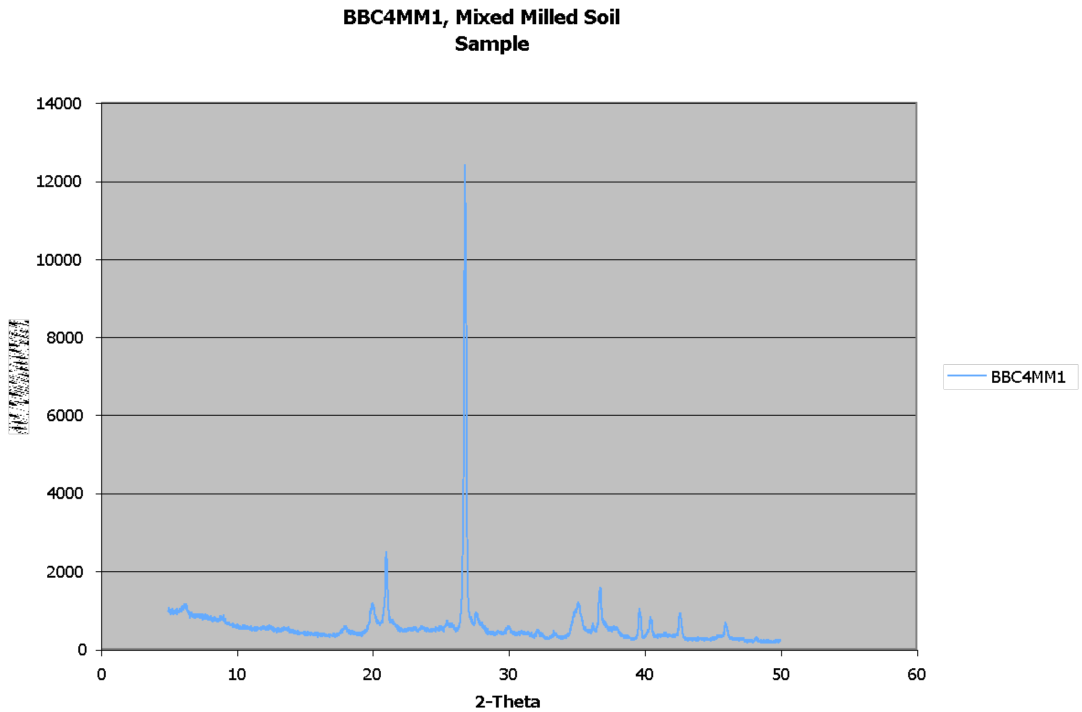
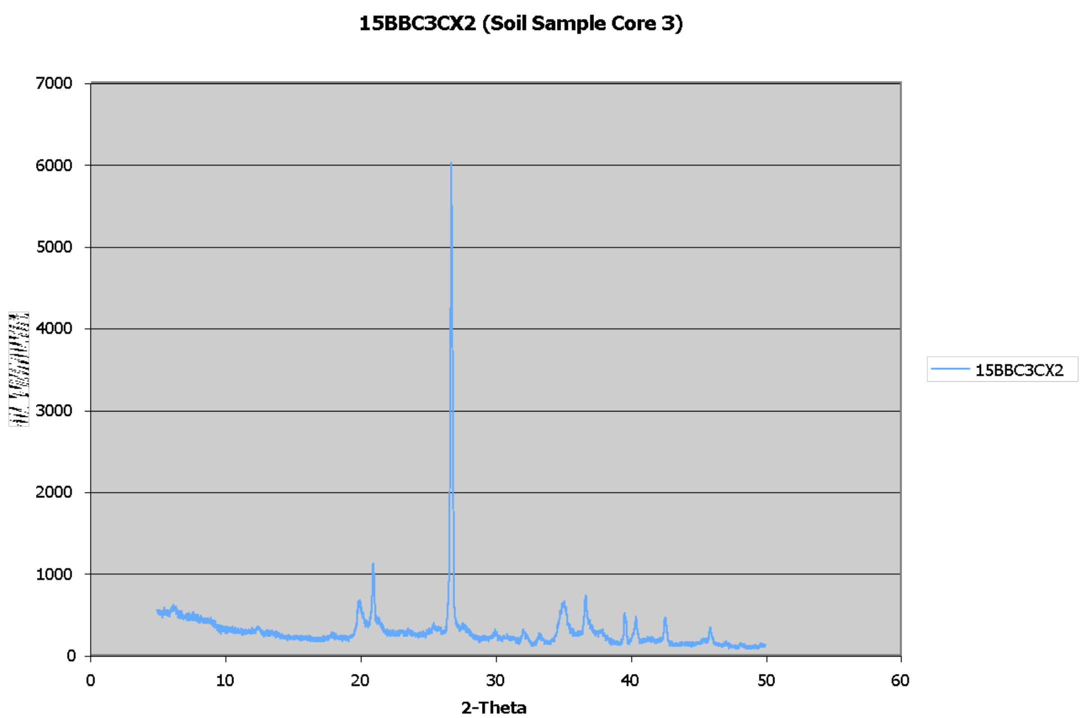
Appendix 4: Sample XRD Scans and Observations

Soils:




BBC4MM1, Mixed Milled Soil Sample
14000
12000
10000
8000
6000
4000
2000
0
0
10
20
30
40
50
60
2-Theta
BBC4MM1


15BBC3CX2 (Soil Sample Core 3)
7000
6000
5000
4000
3000
2000
1000
0
0
10
20
30
40
50
60
2-Theta
15BBC3CX2

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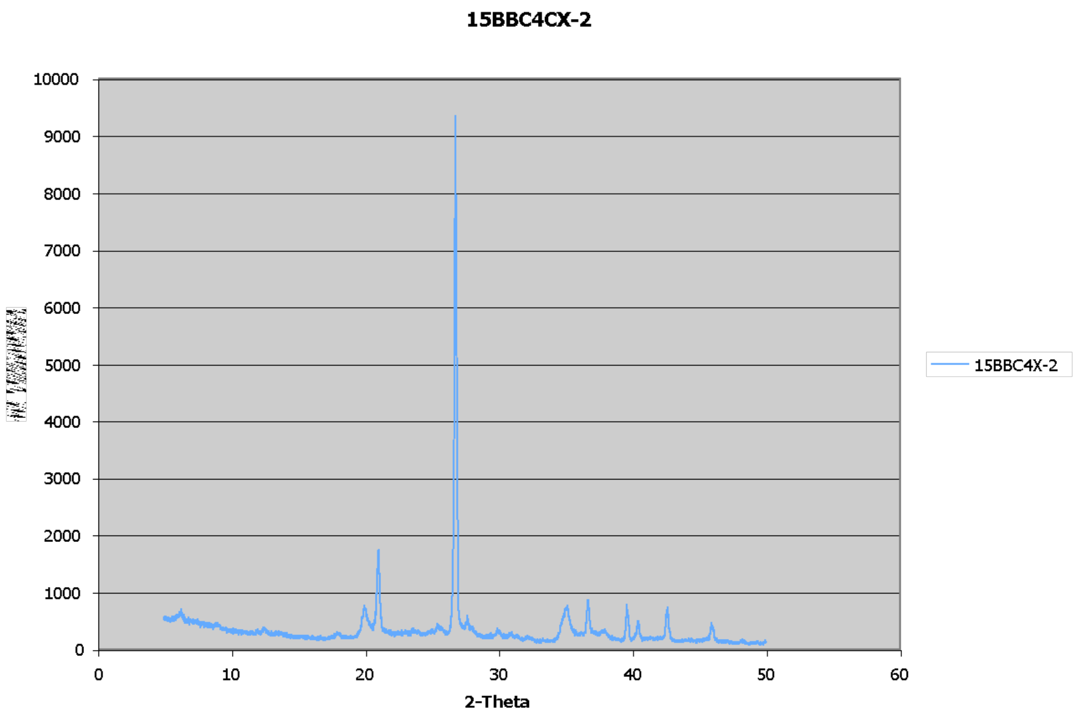

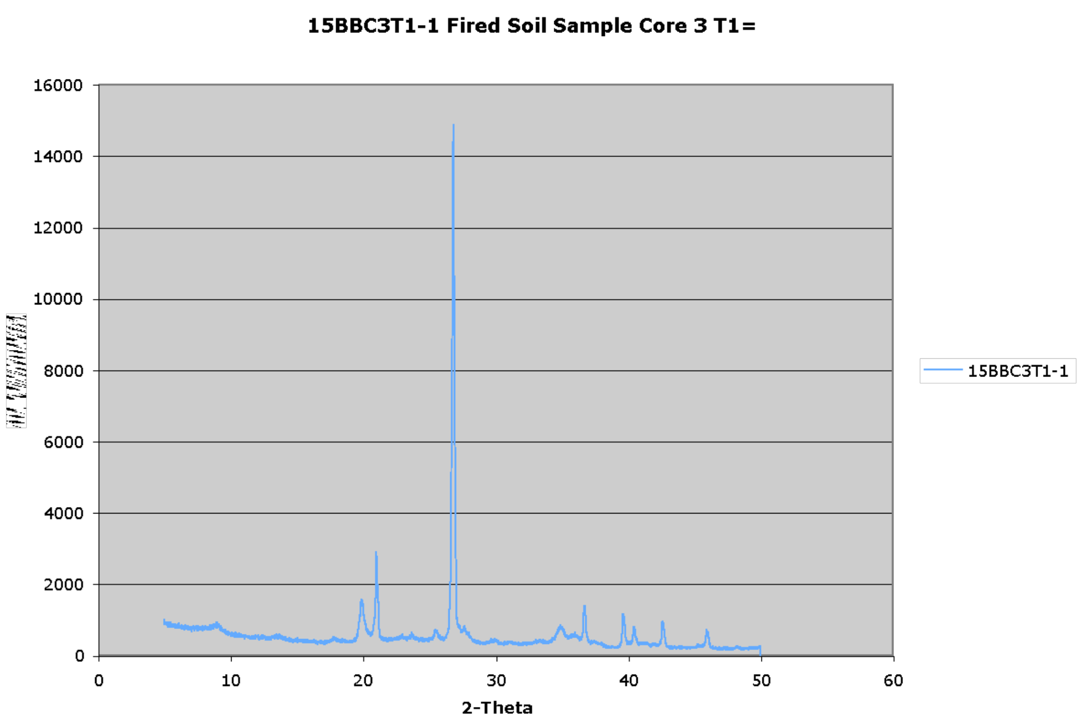

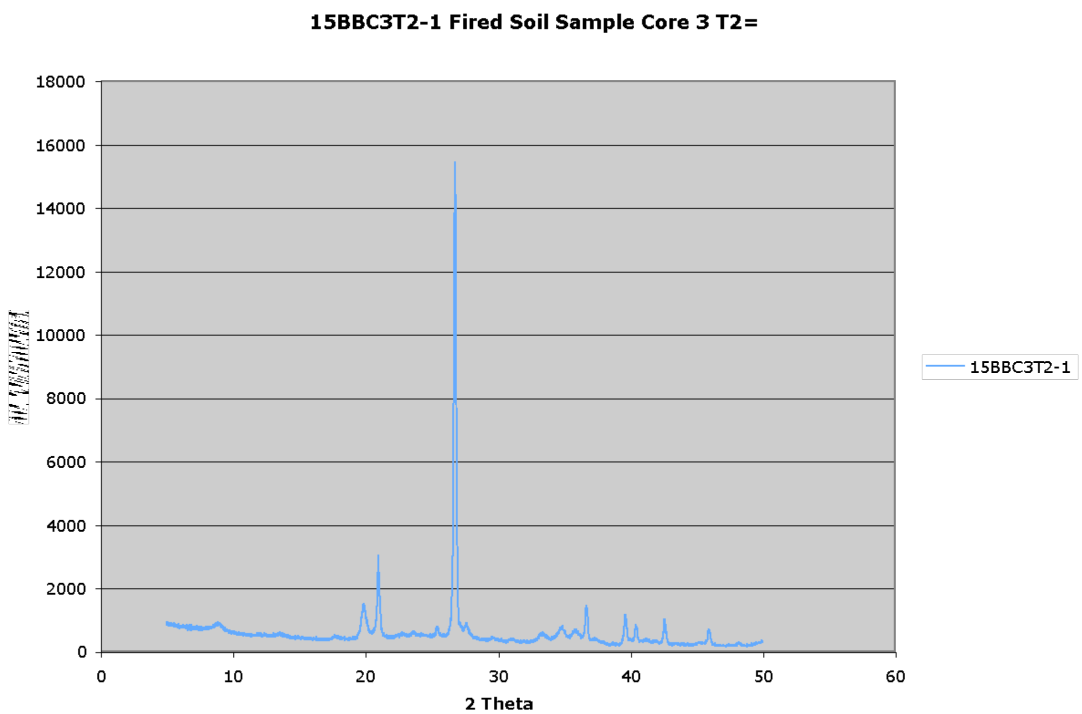
15BBC3T2-1 Fired Soil Sample Core 3 T2=
18000
16000
14000
12000
10000
8000
6000
4000
2000
0
0
10
20
30
40
50
60
2 Theta
15BBC3T2-1

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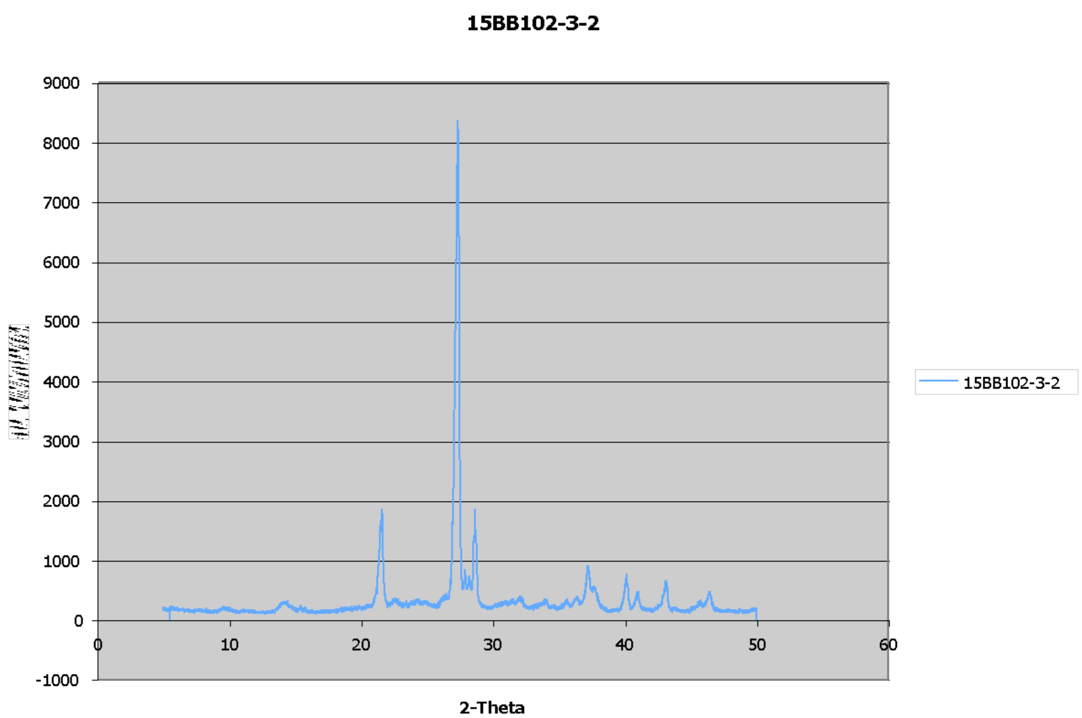
Sherds:

15BB102-3-2
Item Description/Type: waster pit, saggar base, sherd
Color: red-orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3): scans compare most closely to those of T1 (600C) & T2 (838C)
Other Observations: thick (~1 cm) section of a saggar base



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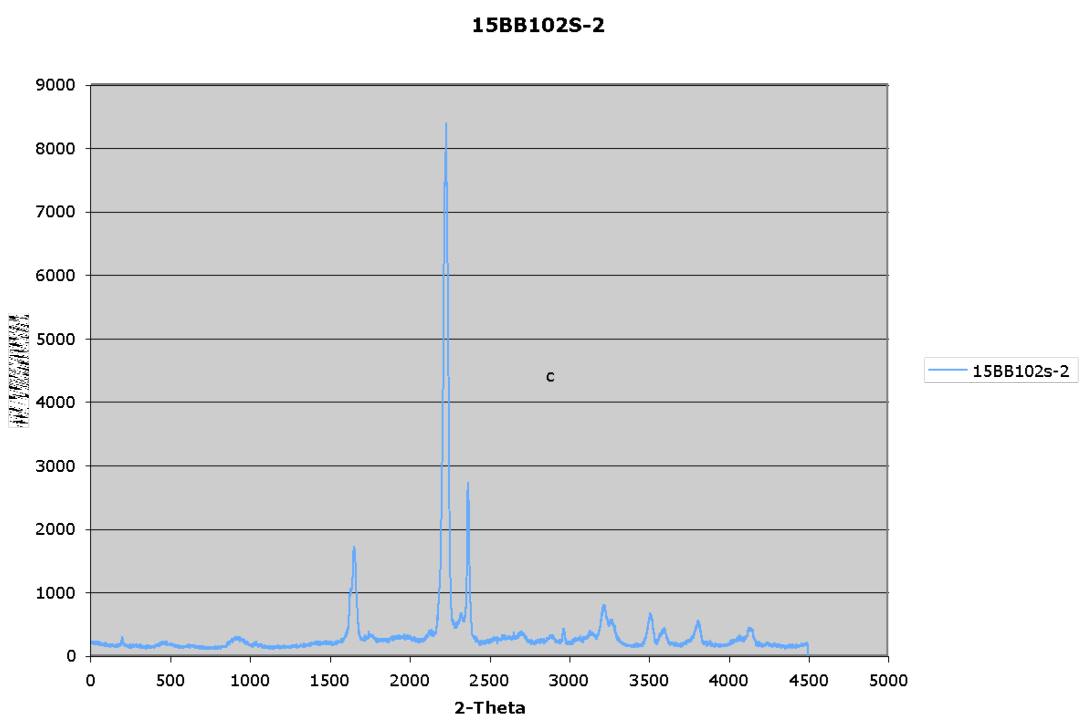
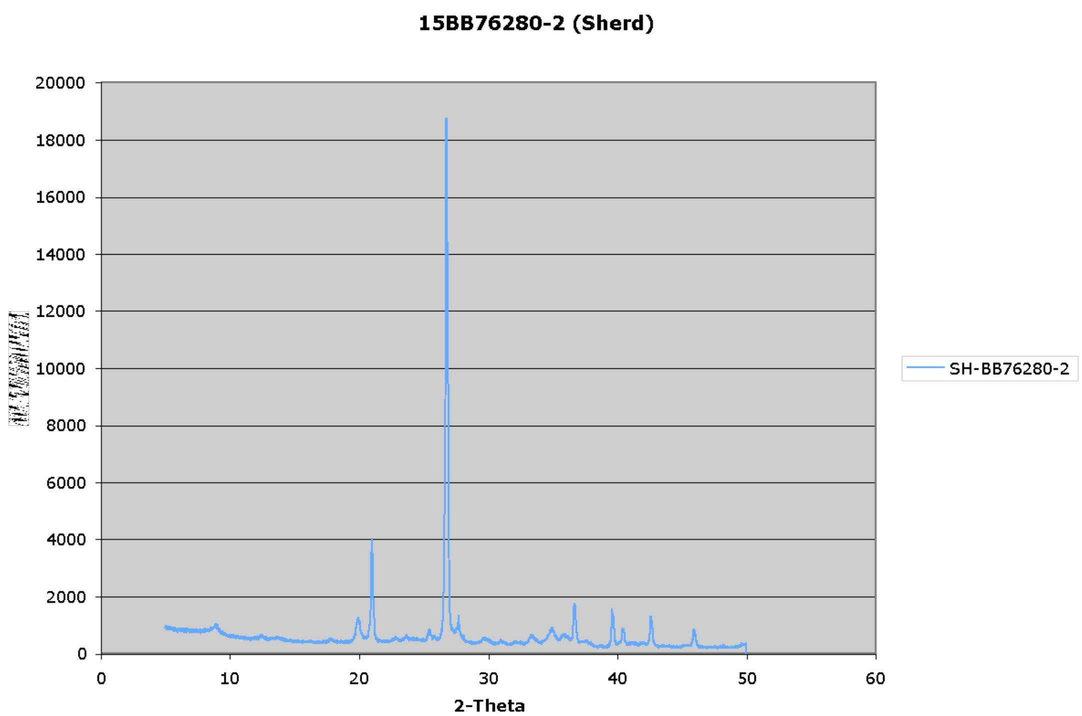
15BB102-S-2
Item Description/Type: waster pit, glazed 2 sides, sherd
Color: red-orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3):

Other Observations: has clear glaze; scan varies and has additional peaks, very similar to 15BB102-3



15BB76-280-2
Item Description/Type: chunks of fired clay
Color: red-orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3):
is closely similar to T2 (838 C)
Other Observations:



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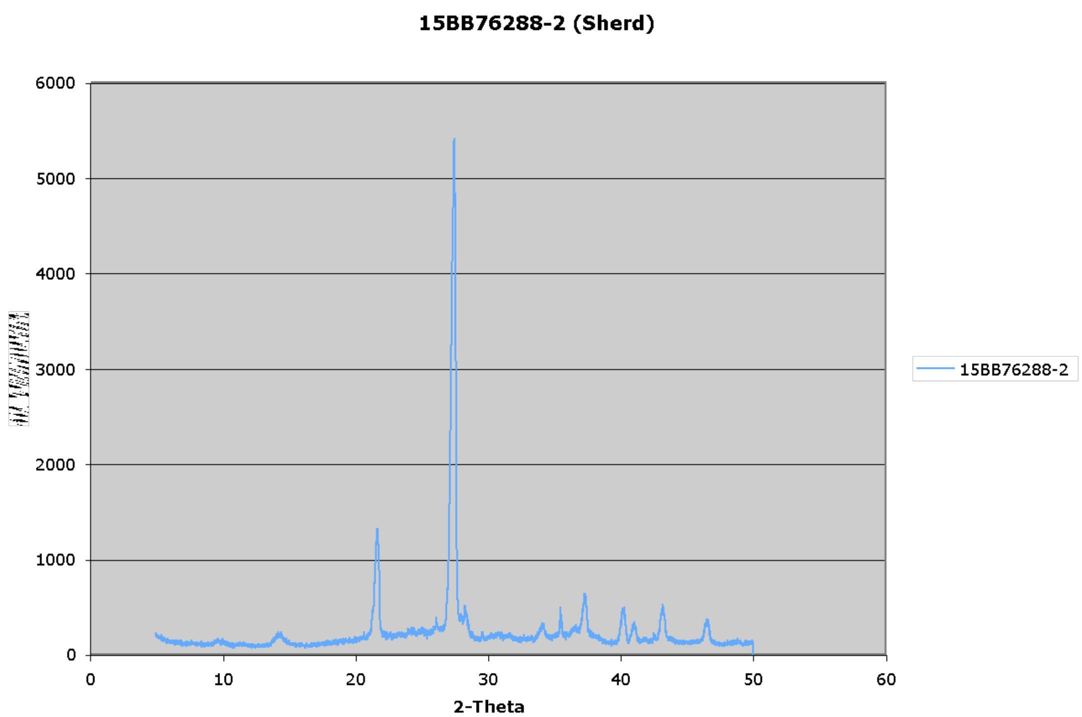
15BB76-288-2

Item Description/Type: glazed one side (crawling, clear glaze)
Color: red orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3):
Most similar to T3 (838 C) & T4 (1220 C)
Other Observations:



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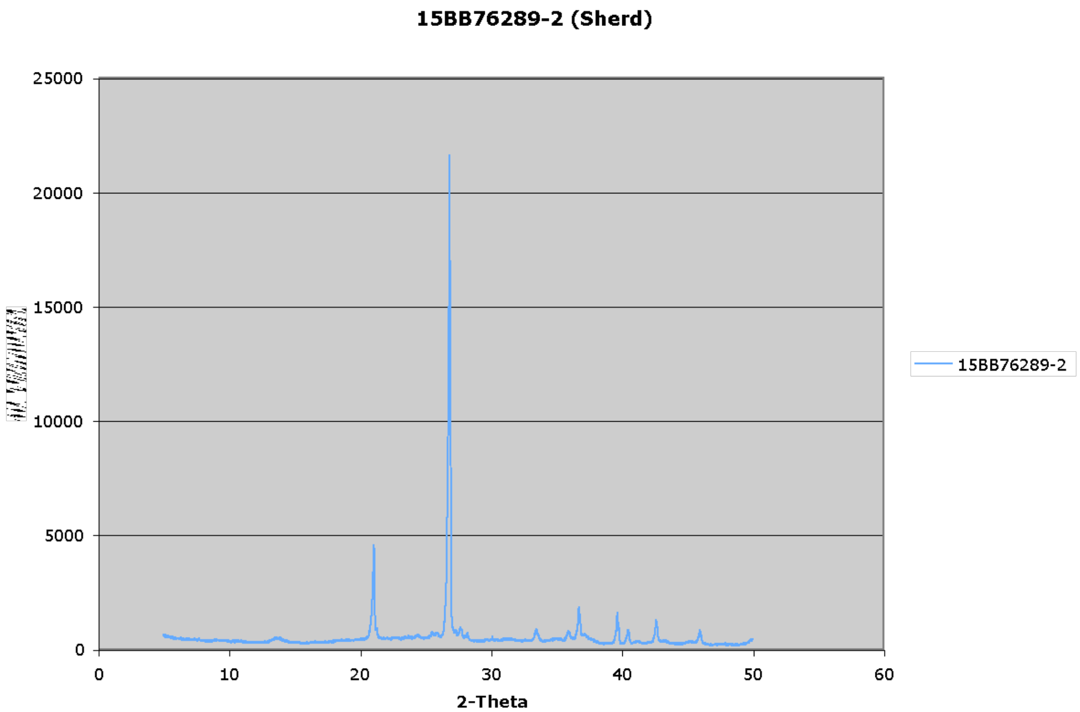
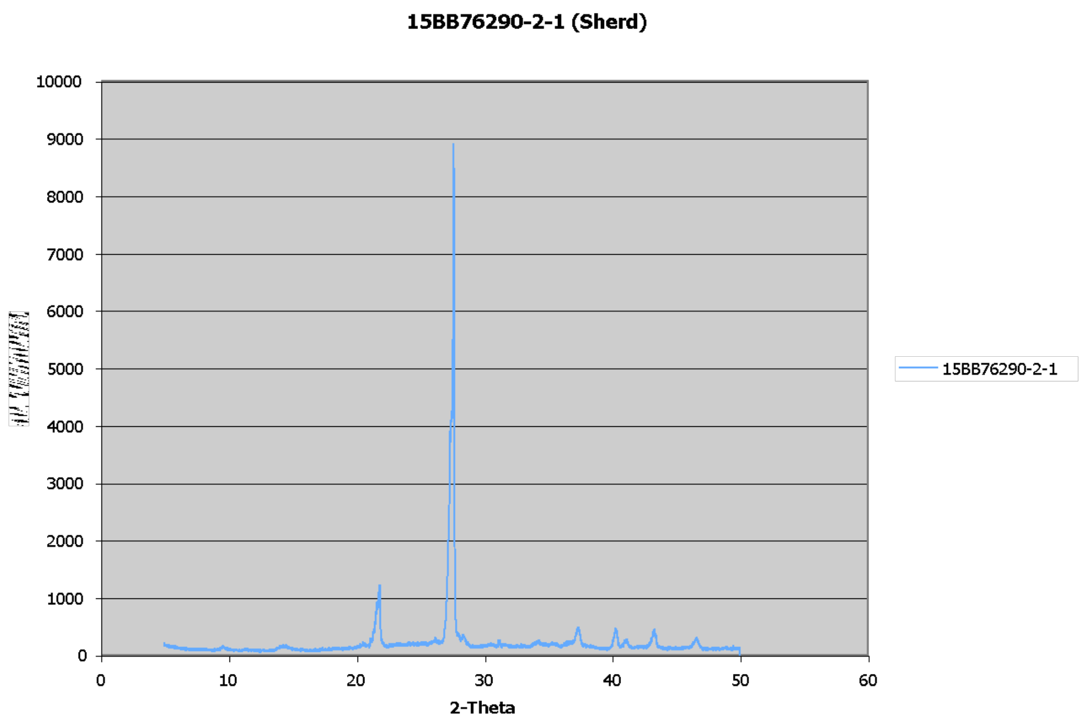
15BB76-289-2
Item Description/Type: unglazed
Color: red orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3): Most similar to T3 (838 C)
Other Observations:



15BB76-290-2-1

Item Description/Type: smooth glaze patchy areas of dark glaze (reduction? Carbon trapping?); glazed both sides
Color: red-orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3):
Most similar to T3 (838 C)
Other Observations:



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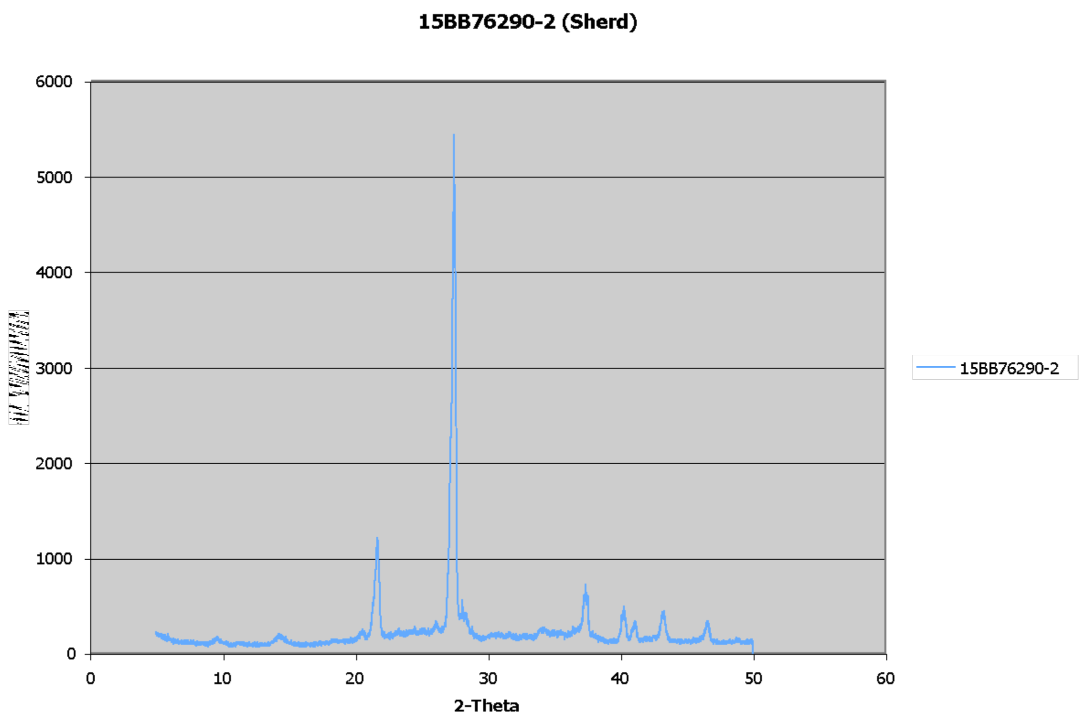
15BB76-290-2

Item Description/Type: smooth glaze patchy areas of dark glaze (reduction? Carbon trapping?); glazed both sides
Color: red-orange
Fragility/Toughness: crushed readily
Comparison to Soil Firing Temperature Comparison (Chart 3):
Most similar to T3 (838 C)
Other Observations: same as 290-2-1



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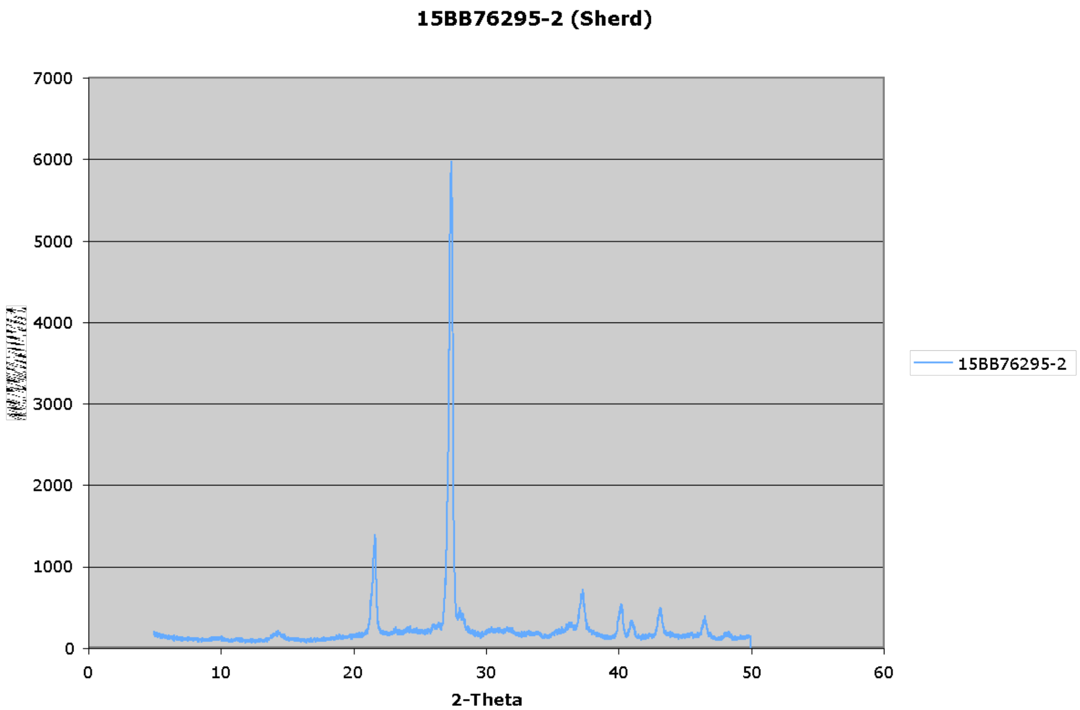
15BB76-295-2
Item Description/Type: labeled “green glaze”; glazed 2 sides
Color: interior clay body very grey to putty grey-brown
Fragility/Toughness: crumbled readily, softer, very fine
Comparison to Soil Firing Temperature Comparison (Chart 3):
Most similar to T1 (600 C)
Other Observations:



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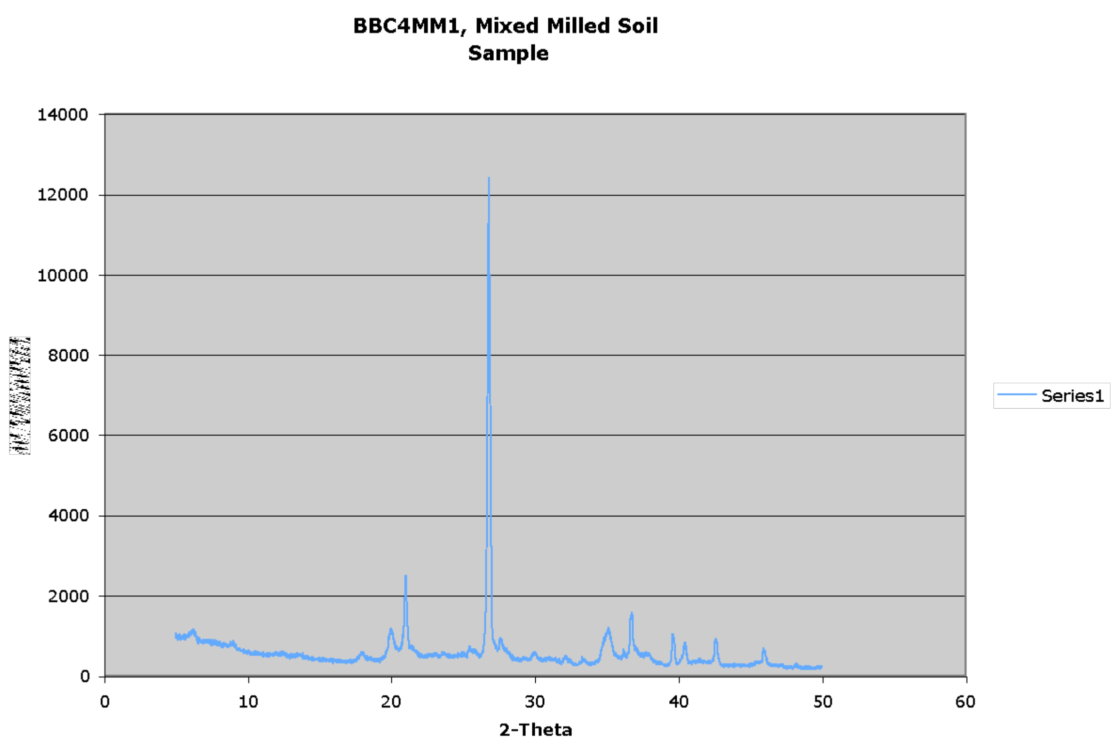
Appendix 5: XRD Comparisons

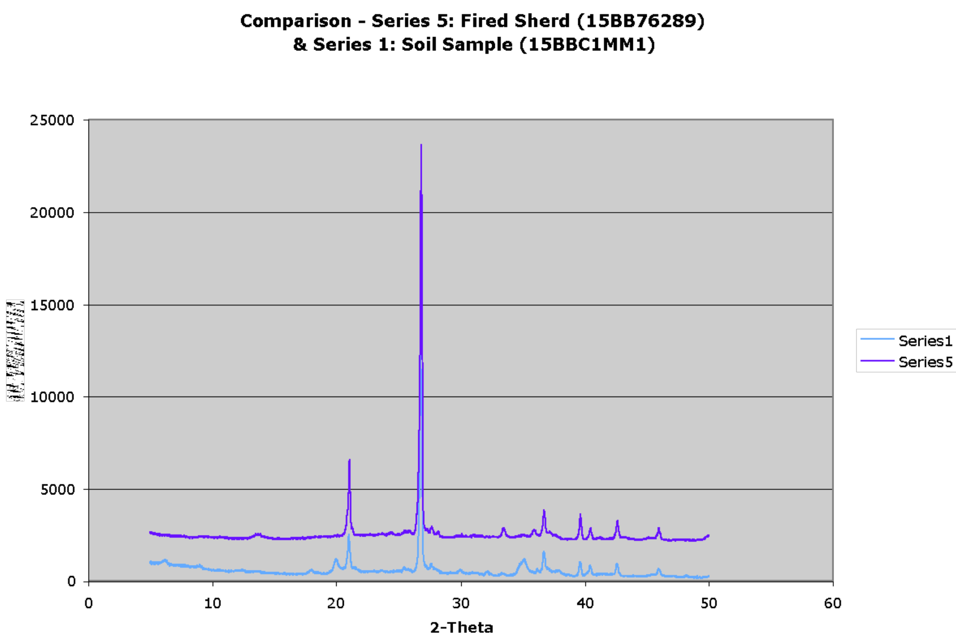
In addition to the samples taken directly from a specific soil height and site coordinates, a series of mixed samples were created. These were mixed together and milled into a smooth, fluid slip before being poured into a plaster tile mold for drying. The dried tile was then ground into a powder for XRD analysis. This extra step was intended to get closer to a similar type of processing to that of the actual preparation of the clays, as the pottery was known to have a mill and would likely have used a blend of material taken from across the range of the basin. The XRD pattern is shown in **#1, below.**

1. Soil Sample after Mixing and Milling



A soil sample is shown in comparison with a sherd sample (15BB76-289) in **#2**, below. While they bear distinct similarities based on the high percentage of silica in each, there are some differences in the smaller peaks that can be attributed to variances in the materials as well as to phase changes that occurred during firing.

2 Comparison ~ Fired Sherd and Unfired Soil Sample



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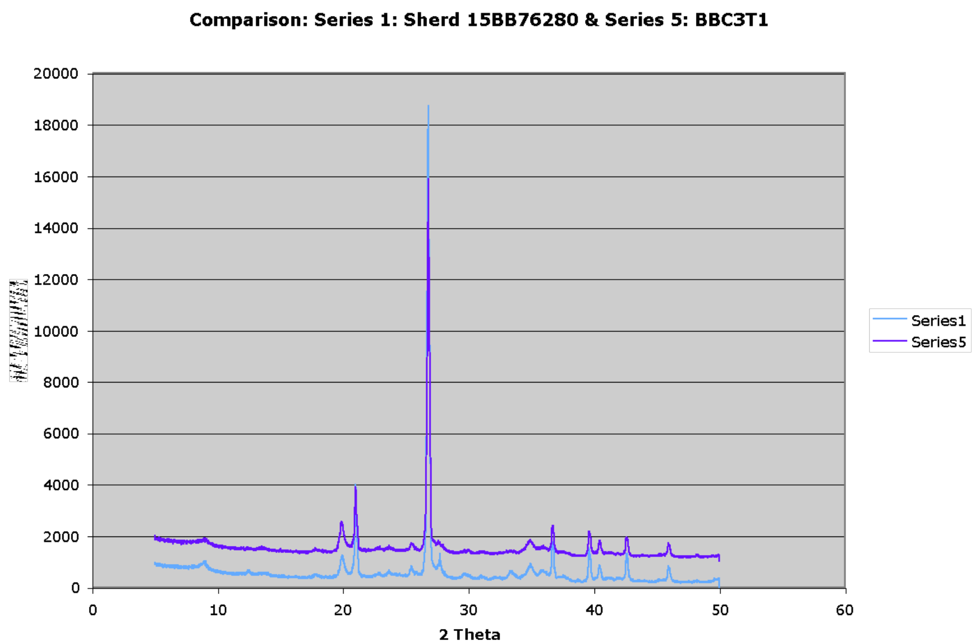
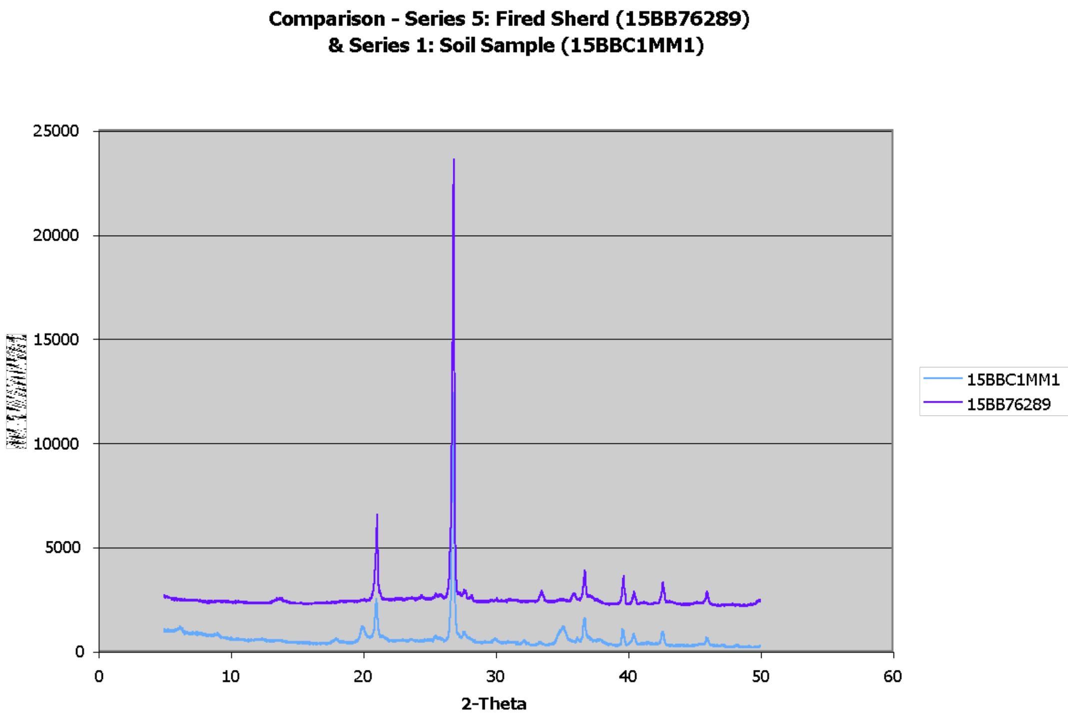
A comparison of a fired soil sample and a representative sherd sample are shown in **#3, below.** Unlike the samples in **#2**, above, the samples in **#3** are more closely similar, which can likely be attributed to having undergone similar firing changes.

3: Comparison: Sherd 1 and Fired Soil Sample



In **#4,** below, an additional fired sherd sample is compared to the same soil sample in **#2**.

4. Comparison: Sherd 2 and Fired Soil Sample



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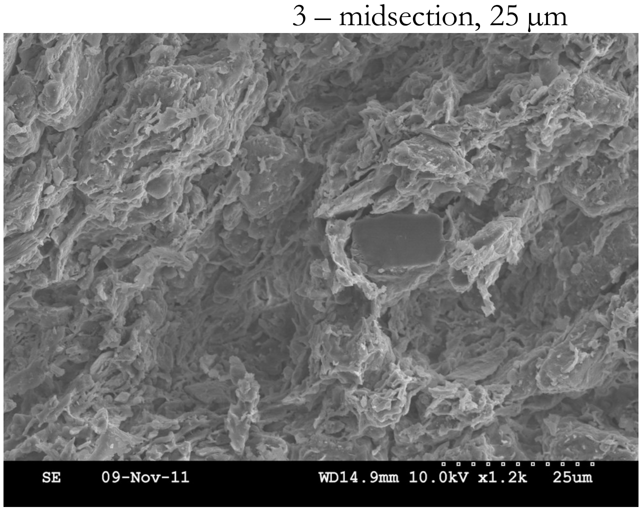
Appendix 6: Scanning Electron Micrography - SEM:

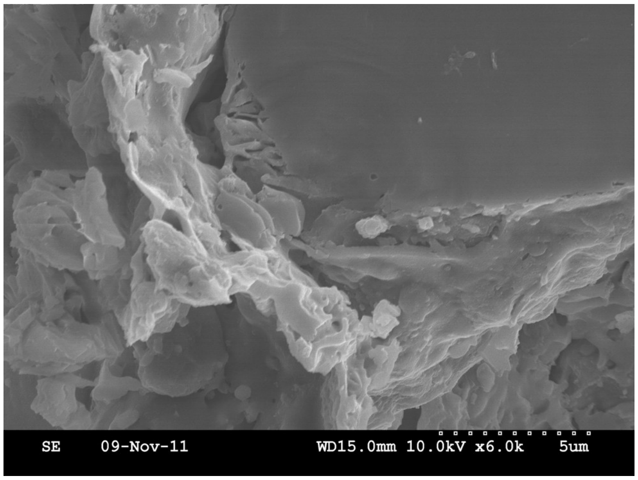
The scanning Electron Micrograph (SEM) images were created using the SEM at the University of Kentucky materials department. Images were taken from the midsection of sample 15BB295 at 25 μm and 5 μm, while the glazed edge is shown at 50 μm, 10 μm, and 5 μm. In the unglazed sample 15BB289, images were taken at 50 μm, 2 μm, and 1μm. (See Figures 3 – 11, Appendix, below.) These images are provided for reference, but EDS analysis was not undertaken due to cost and time factors. This type of testing would provide additional information about the elemental composition of the clay body.

Figures 3 – 7: SEM Sample Site No. 15BB295:

3 – midsection, 25 μm

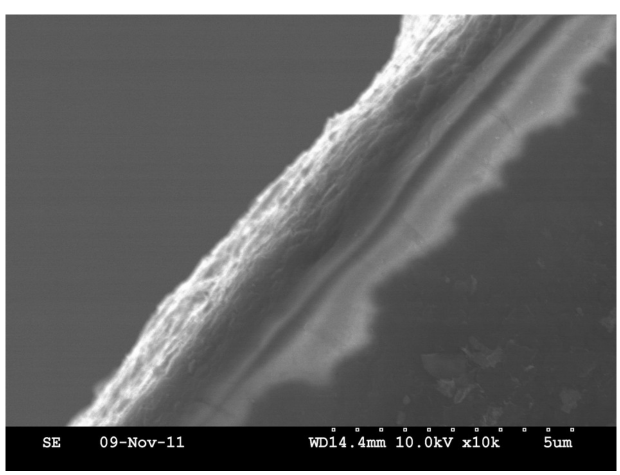


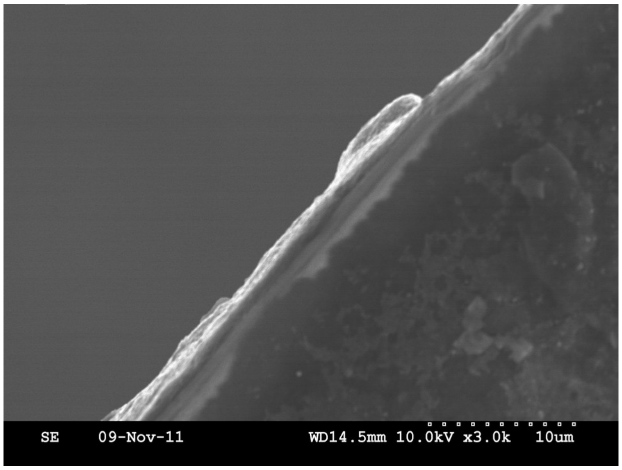
4 – midsection, 5 μm



5 – glazed edge, 50 μm

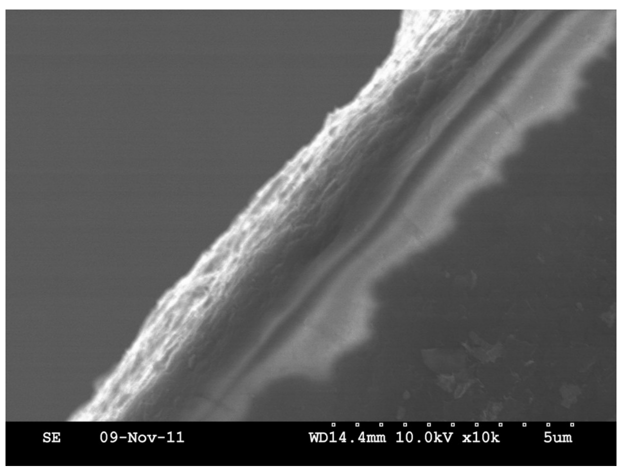
6 – glazed edge, 10 μm



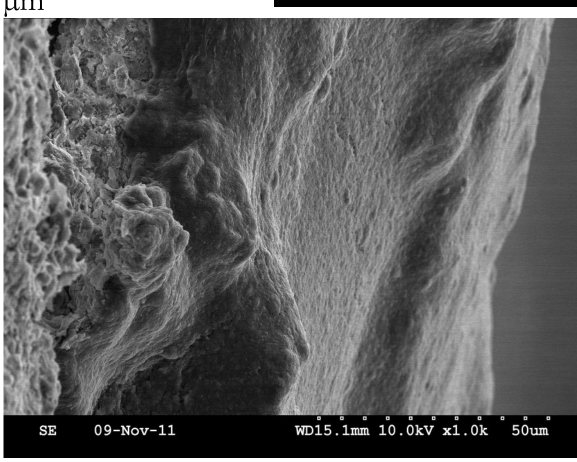


7 – glazed edge 5 μm

Figures 8 – 11: Sample No. 15BB289



8 – raw edge, 50 μm



9 – raw edge, 2 μm

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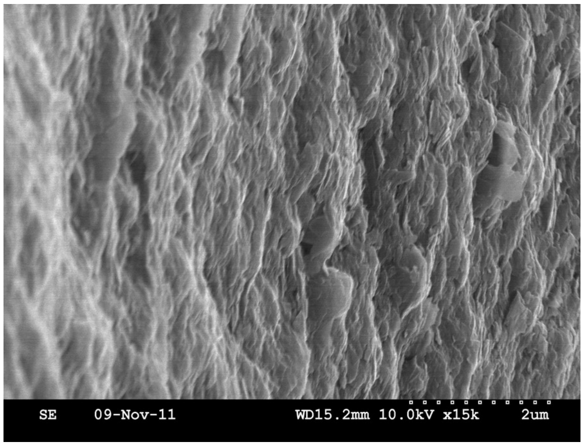
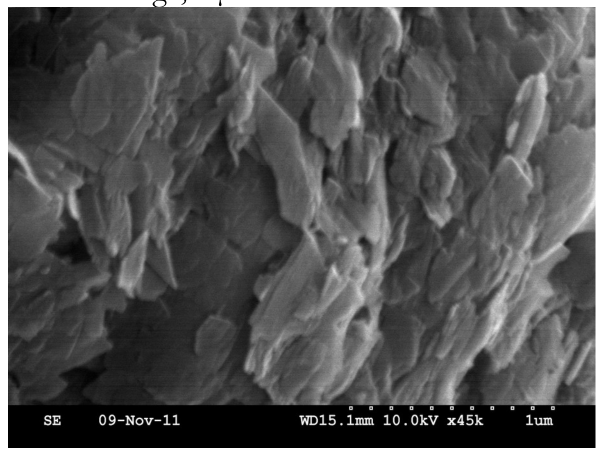


10 – raw edge, 1 μm



11 – raw edge, 2 μm