

Naveed Usman *
Shakirullah **
Rashid Ahmad ***

Microstructural Analysis Of Potsherds From Archaeological Explorations Of The Kaghan Valley, Mansehra, Pakistan

Abstract

Investigations on the pottery sherds using Scanning Electron Microscope along with the Energy Dispersive X-rays is a strong tool that give characteristic information about the mineralogical composition and percentage of the minerals in the fired pottery sherds. It is possible to establish the nature of pottery sherds from the micrographs obtained. The size and distribution of the particle size as well as the pores are not uniform which may be the result of variations in the firing temperatures during the manufacturing process. The results show that the pottery samples selected for examination in the current study were fired between the temperature ranges of 800-1050°C.

Key Words: Microstructure, Firing Temperature, SEM-EDX, Micrograph, Sintering

1. Introduction

Potsherds collected from the surface of different archaeological sites of the Kaghan Valley have been analyzed to trace the source, distribution, technology and firing temperatures. The area under study has remained veiled from the archaeological scientists to find out the culture, technology and life style of the people lived at the time of its manufacturing. The comparison of the elemental ratio in specimen with the established geological features of the area has given a reliable information about the possible raw material, place of manufacturing, clay (calcareous or non-calcareous) and distribution, while the images from Scanning Electron Microscope have given information about the surface morphology, firing temperature range and the steps involved in the firing process. The surface morphology also gives an idea about the firing technology used by the potters.

* PhD Research Scholar, Department of Archaeology, Hazara University Mansehra, Pakistan, Email: naveedusman2@gmail.com

** Associate Professor, Department of Archaeology, Hazara University Mansehra, Pakistan, Email: shakir@hu.edu.pk.

*** Professor, Department of Chemistry, University of Malakand, Dir (Lower), Pakistan, Email: rashidahmad@uom.edu.pk.

2. Material and Methods

Four pottery sherds have been selected from four different areas and given codes (Table 1; Pl. 1). The physical and chemical characterization of the ceramic artifacts in terms of mineralogical composition provides significant information about the technology, firing techniques and firing temperatures during the manufacturing (Palanivel et al., 2009).

The technique and instruments used for the present study are Scanning Electron Microscope (SEM) equipped with the Energy Dispersive Spectrum (EDX or EDS) micro-analysis detector. This combination has found very useful for this kind of pottery analysis (Tite, 1992). It is a non-destructive technique for the elemental analysis which leads to the characterization of the pottery. It measures the whole X-ray spectrum and provides a quick determination of elemental composition in the targeted specimen (Froh, 2004).

Specimens of the potsherds collected from four different archaeological sites have been selected for the analysis. Physical characteristics such as type, color, surface texture, decoration etc. have been found and then subjected to the Scanning Electron Microscope imaging with magnification of X2000 to find out the pattern of surface morphology. The surface morphology images give an idea of the possible firing temperatures and the total number of steps involved in the firing process (single step firing or multi-step firing). The EDX diffractogram has been obtained by standardizing it for all element analysis at 20000 KeV. The diffractogram gives the elemental composition in the each specimen in terms of the atomic and weight percentage of each element which, on comparison with the established geological features, gives indications towards establishing an idea about whether the pottery has been locally produced or imported from somewhere else. It also gives a reliable data in terms of elemental percentage that if the pottery produced from a single source of raw material has been distributed among all the four sites which, as a long term outcome, give an idea about the cultural migration, trade routes and diversity of the culture in the area.

2.1. Geological Features of the Selected Sites

The selected sites of Ganool (GNL), Shogran (SGN), Ratta Nulla (RTN) and Tarla Paror (TRP) lie in the Lower Kaghan Valley range from Balakot to Mahandri. The main geological formation in the region is Salkhala Formation (Paul et al., 1988). The Salkhala Formation mostly consists of the quartz Schist, Marble, Graphite Schist, and Quartzo-feldspathic Gneiss. The large proportion of the formation consists of quartz schist, which is mainly fine grained mica chlorite quartz schist. 20 to 40% of the schist consists of chlorite and muscovite while the remainder is quartz (James et al., 1975).

The geological features show that the local raw material contains C, O, Na, Mg, Al, Si, K, Ca, Fe etc.

3. Results and Discussion

The characteristic X-ray energy which is emitted when a solid specimen is struck by the electrons enabled to identify the main elements that were present in the pottery samples (Feathers et al. 2006: 89-133). The EDX spectra of the four sherds are shown in the Fig. 2 – Fig. 5 respectively and the weight and atomic percentages of each element is presented in Table 3.

The percentages of the constituent elements present in the specimen give very interesting information about the nature of clay used and the provenance. Both the weight and atomic percentages show that carbon is found only in the sherd collected from Ganool Site (GNL-1). The oxygen is almost same in all the samples. Sodium is in minute quantity in all the samples having exactly same atomic percentage in the samples SGN-2 and RTN-1, which shows that if sodium is taken as the reference element, the sherds from Shogran and Ratta Nulla are manufactured from the same material. Magnesium has varied atomic percentages in all the samples and found the most in RTN-1 showing that the raw material might contain some dolomite or Epsom.

The weight percentage of Aluminum is exactly same in the sherds GNL-1 and RTN-1 (0.02%) while that of SGN-2 and TRP-1 have the same (0.01%). If Aluminum is the reference element then the raw material of sherd from Ganool resembles with the raw material taken for the production of sherds from Ratta Nulla and similarly, that of Shogran resembles with Tarla Paror and same is the situation with the percentage of Silicon (0.04% in GNL-1 and RTN-1 while 0.03% in SGN-2 and TRP-1). Comparatively greater percentage of potassium is found in the sherd RTN-1 than all other sherds while the percentage of calcium is greater in TRP-1 than all other sherds.

The weight percentage of iron is exactly same in all the potsherds (0.02%). Now if, iron is taken as the reference element, all the sherds collected from four different sites are produced from the same raw material. It may be assumed that either the percentage of iron is same in the entire region geologically or the pottery from the same kiln has been distributed in all the selected sites. Minute atomic percentage of Nickel is found in all the three pottery sherds except TRP-1, while relatively greater percentage of Cadmium is found in the sherd TRP-1 than RTN-1 and is absent in the other two sherds i.e. GNL-1 and SGN-2.

The presence of Calcium in the sherds indicates that the pottery was fired at relatively low temperature. The pottery sherds have less than 6% of the calcium

content shows that the clay used for pottery making was non-calcareous in nature (Maniatis and Tite, 1981).

The scanning electron micrographs of the selected sherds show irregularity in shapes and sizes of the constituent particles. The potsherds surfaces are comprised of plate like and somewhere needle like grains with the crystallite size of different micrometers which gives an idea that the pottery was fired at relatively low temperature which caused heterogeneity (Krapukaitypte et al. 2006: 383-388). The relatively smooth surface of the sherd TRP-1 may be due to reflective nature caused by the early compacting of the material by heating or may have happened naturally in the mineral deposits which is called as sintering (Rye, 1981).

The micrographs also show some smooth surface spots which reveal the initial vitrification stage for all the sherds under investigation. It is therefore considered that the firing temperature for the pottery production was ranging in between 800 to 850°C (Minitias and Tite, 1981).

The micrograph of RTN-1, if carefully examined, reveals that the some layered particles were shaped with a number of incisions. The cubical shaped alignment with encompassing distribution of the pores gives an idea that the sherd specimen was fired in more than one step in which the temperature was increasing step by step (Colomban et al., 2004). The presence of fine pores of lesser diameter may have been caused due to continuous vitrification in the reduced atmosphere (J. Froh 2004: 159-176). From the formation of micro pores in the specimen RTN-1 it may be assumed that the sample may have been fired at the temperature below 1050°C.

4. Conclusion

The study of potsherds, numbered as GNL-1, SGN-2, RTN-1 and TRP-1, collected from four different areas of the Kaghan Valley analyzed through SEM equipped with EDX showed a variety of elemental composition and morphology. The elemental analysis by EDX allowed the samples to be grouped on the basis of weight percentage and atomic percentage. From the elemental data and geological features of the area, it can be concluded that the pottery was locally made but the raw material or the pottery, after its production may have been migrated or traded among the sites under discussion. It can be concluded that there must have been some central kiln(s) in the entire area in which the pottery was produced locally without any specific pre-established firing technology. From this central kiln(s) the pottery was distributed/traded in the entire area. The SEM patterns of the samples suggest that the pottery sherds GNL-1, SGN-2 and TRP-1 have been fired in the temperature range between 800 – 850°C, while the sherd number RTN-1, as discussed above, has been fired in the temperature range of 1000 – 1050°C.

5. Acknowledgements

The under discussion material has been collected during the exploration of the area under project entitled “Archaeological Survey of Un-Explored Parts of Districts Mansehra and Haripur of Hazara Division” funded by Ministry of Information, Broadcasting and National Heritage, Govt. of Pakistan.

We acknowledge the support from the National Research Laboratory, National Textile University Faisalabad, for analysis of potsherds.

Table 1: Selected Sites and Potsherds

S. No.	Name of the Area	Site Code	Potsherd Number
1.	Ganool	GNL	GNL-1
2.	Shogran	SGN	SGN-2
3.	Ratta Nulla	RTN	RTN-1
4.	Tarla Paror	TRP	TRP-1

Table 2: Physical Attributes of the pottery sherds selected for the study

S. NO	No.	Site	Type	Thickness (mm)		Color (Munsell Color System)		Surface	Decor ation
				Min	Max	In	Out		
1	GNL -1	Ganool	Body Sherd	8	11	10YR 2/2 highly dark brown	7.5 YR 5/5 brown	Burnished	No
2	SGN -2	Shogran	Body sherd	9	10	7.5YR 6/3 light brown	10YR 1 6/2 light brownish grey	Burnished	No
3	RTN -1	Ratta Nulla	Base Fragment	14 Base Diameter: 64	18	10YR 1 6/2 light brownish grey	10YR 5/2 grayish brown	Burnished	No
4	TRP-1	Tarla Paror	Rim Fragment	10	12	10YR 2/2 highly dark brown	10YR 2/2 highly dark brown	Burnished	No

Table 3: Elements present in the potsherds

S. No.	Element	Elemental Concentration (Weight %)				Elemental Concentration (Atomic %)			
		GNL-1	SGN-2	RTN-1	TRP-1	GNL-1	SGN-2	RTN-1	TRP-1
1.	Carbon (C)	0.01	-----	-----	-----	7.42	-----	-----	-----
2.	Oxygen (O)	0.07	0.05	0.06	0.04	56.18	59.00	55.56	53.47
3.	Sodium (Na)	0.00	0.00	0.00	0.00	0.63	0.96	0.96	0.27
4.	Magnesium (Mg)	0.00	0.00	0.00	0.00	1.76	1.27	2.07	1.96
5.	Aluminum (Al)	0.02	0.01	0.02	0.01	8.38	8.63	9.48	10.68
6.	Silicon (Si)	0.04	0.03	0.04	0.03	19.47	21.94	21.28	23.44
7.	Potassium (K)	0.00	0.00	0.01	0.00	0.69	2.02	2.35	1.98
8.	Calcium (Ca)	0.00	0.00	0.00	0.00	0.92	0.53	0.48	1.41
9.	Iron (Fe)	0.02	0.02	0.02	0.02	3.90	5.54	6.54	6.33
10.	Nickel (Ni)	0.00	0.00	0.00	-----	0.66	0.11	1.04	-----
11.	Cadmium (Cd)	-----	-----	0.00	0.46	-----	-----	0.23	0.46
12.									



GNL-1



SGN-2



RTN-1



TRP-1

Pl. I (Kaghan Valley Mansehra): Selected potsherds for the analysis

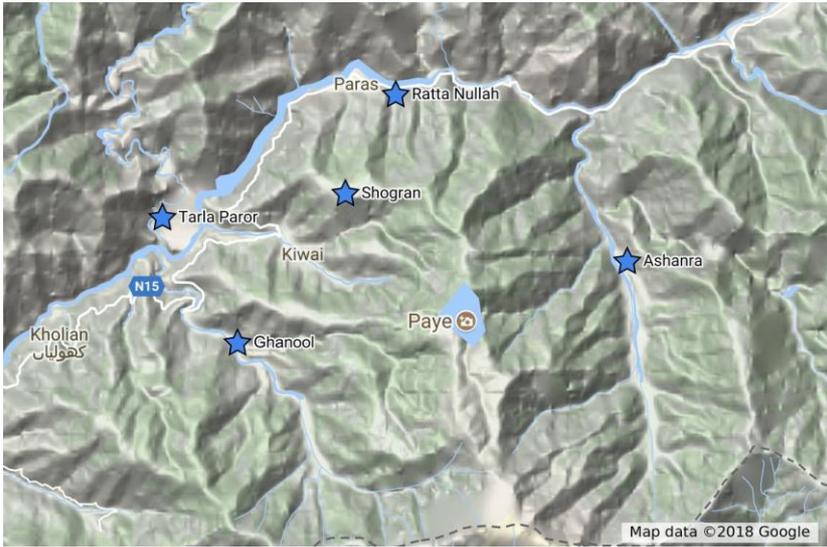


Fig. 1: The Geological Map showing the areas of study

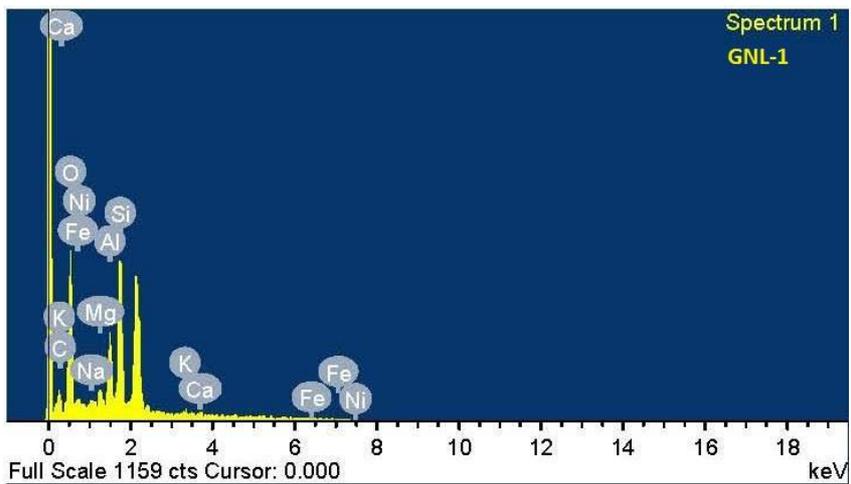


Fig. 2: EDX Spectrum of the sherd GNL-1

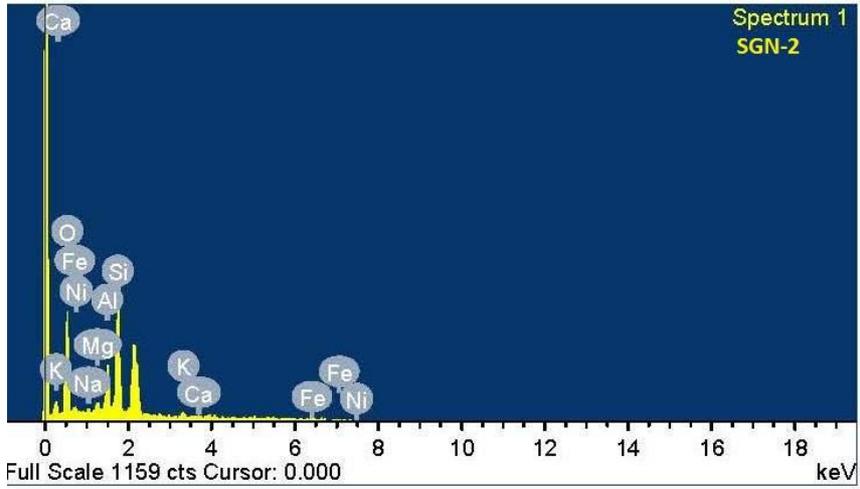


Fig. 3: EDX Spectrum of the sherd SGN-2

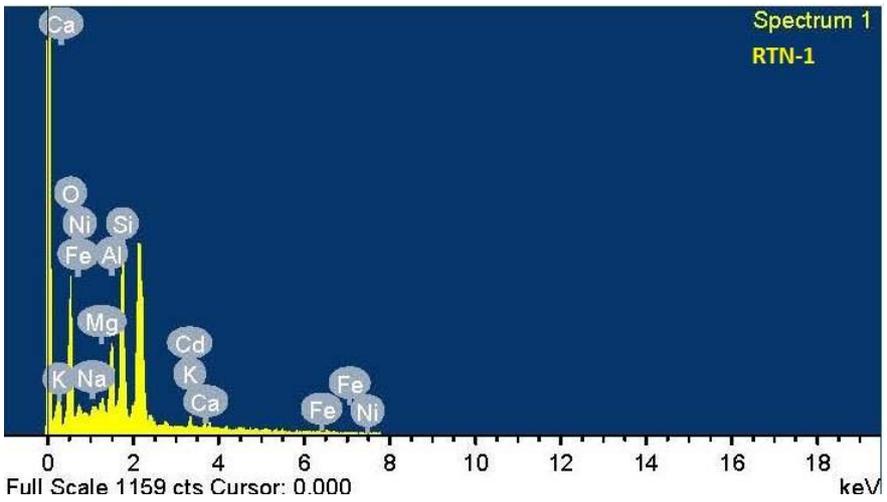


Fig. 4: EDX Spectrum of the sherd RTN-1

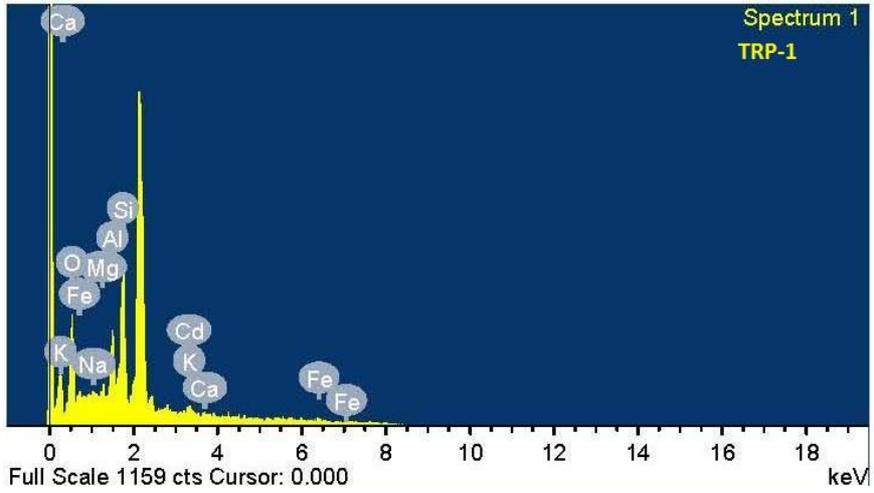


Fig. 5: EDX Spectrum of the sherd TRP-1

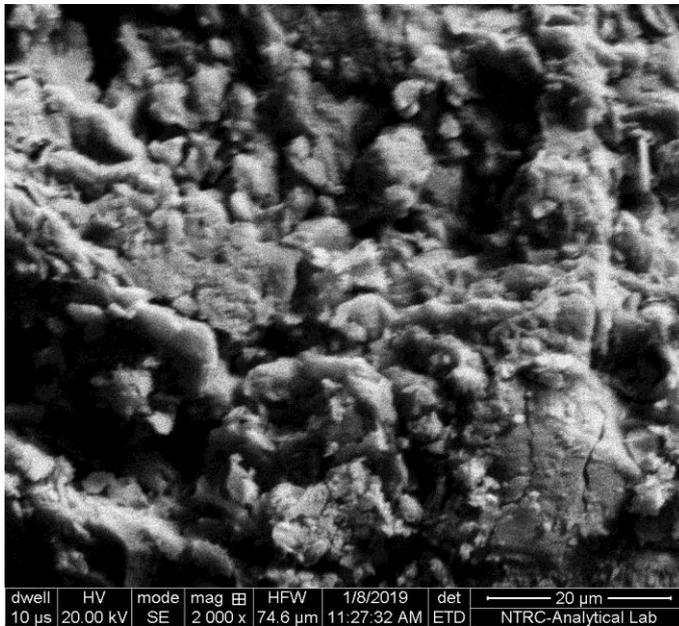


Fig. 6: SEM Micrograph of the sherd GNL-1

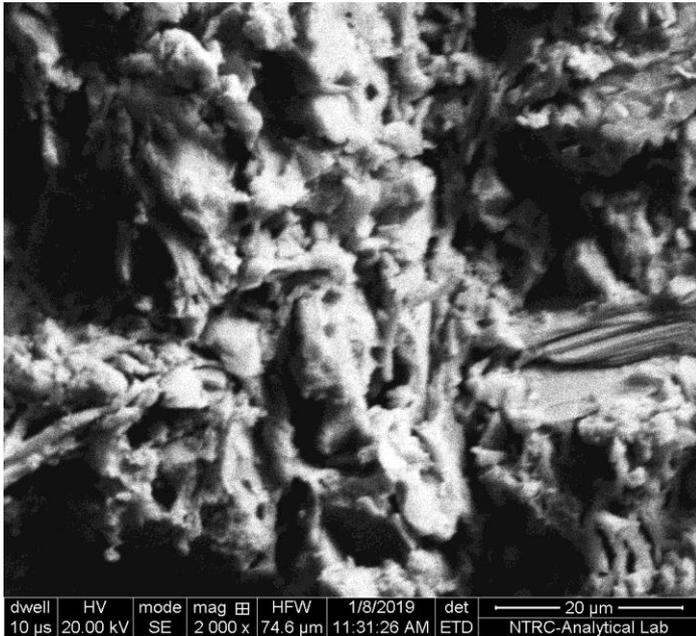


Fig. 7: SEM Micrograph of the sherd GNL-1

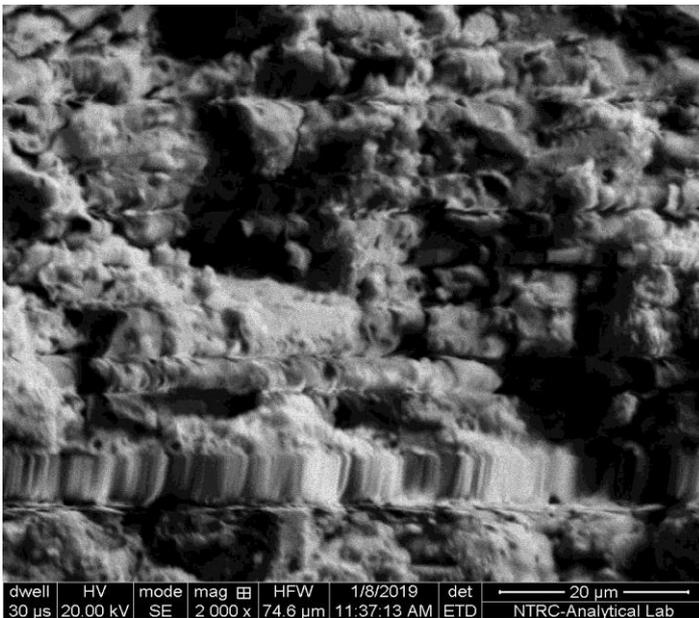


Fig. 8: SEM Micrograph of the sherd GNL-1

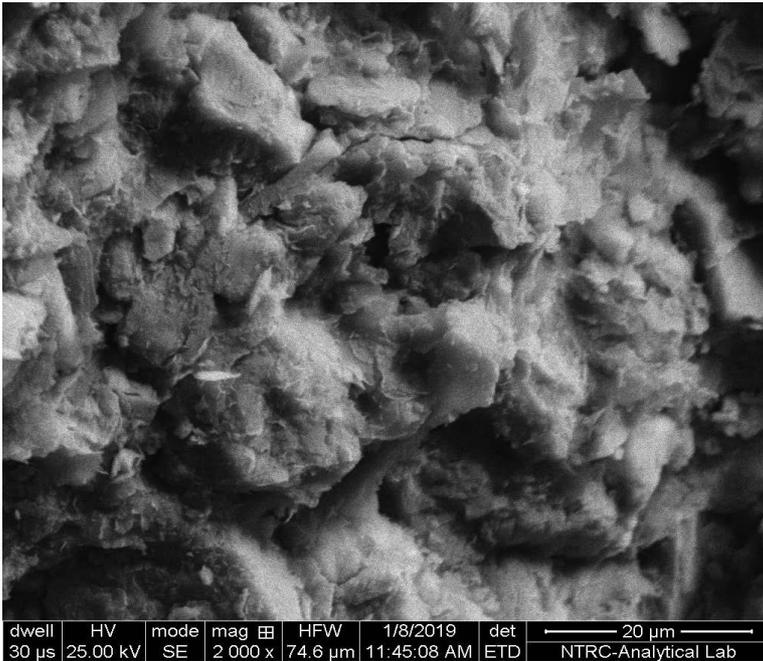


Fig. 9: SEM Micrograph of the sherd GNL-1

Notes & References

1. Bossart, P., Dietrich, D., Greco, A., Ottiger, R., Ramsey, J. G., 1988. The Tectonic Structure of the Hazara-Kashmir Syntaxis, Southern Himalayas, Pakistan. *Tectonics*, 7(2), 273-297.
2. Colombari, P. D. N. Khoi, N. Q. Liem, C. Roche, G. Sagon., 2004. Sa Huynh and Cham Potteries: Microstructure and Likely Processing. *Journal of Cultural Heritage*, 5(2), 149-155.
3. Feathers, K. J., 2006. Explaining Shell-Tempered Pottery in Prehistoric Eastern North America. *Journal of Archaeological Method and Theory*, 13(2), 89-133.
4. Froh, J., 2004. Archaeological Ceramics Studied by Scanning Electron Microscopy. *Hyperfine Interactions*, 154, 159-176.
5. James, A. T., Offield, W., Abdullah, S. K. M., Ali, S. T., 1975. *Geology of the Southern Himalaya in Hazara, Pakistan, and Adjacent Areas* (pp. 6-7). Washington D. C: US Department of State.
6. Krapukaityte, A., Pakutinskien, J., Tantkus, S., Kareviva, K., 2006. SEM and EDX characterization of ancient pottery. *Lithuanian Journal of Physics*, 46(3), 383-388.
7. Maniatis, Y., Tite, M. S., 1981. The Technology Examination of Neolithic-Bronze Age Pottery from Central and Southeast Europe and from the Near East. *Journal of Archaeological Science*, 8, 59-76.
8. Palanivel, R., Meyvel, S., 2009. Microstructural and Microanalytical Study-(SEM) of Archaeological Pottery Artefacts. *Rom. Journ. Phys.*, 55(3-4), 333-341.
9. Rye, O. S., 1981. *Pottery Technology Tarazacum*. Washington. DC.
10. Tite, M. S., 1992. The Impact of Electron Microscopy on Ceramic Studies. *Proceedings of the British Academy*, 22, 111-131.