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Metallurgical Studies on Samples from Central Zagros, Northern Kuhdasht

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Abstract: During the surveys to 2014-2019, Northern Kuhdasht county was studied with the aim of identifying the potential of ancient metalwork and the existence of sites related to the metalworking activities. These Surveys have yielded eighty sites containing slag and or possible places related to industrial activities. In this research, the physical and chemical properties of slags recovered from ten places in four sites, namely Changari, Chalghesholeh, Sargiz, and Dalab, have been studied. The main purpose of this study has been to identify the type of smelting and melting techniques used in the past. For this aim, petrographic and elemental analyses have been done on the slag samples to investigate the mineralogical and chemical compositions.

Keywords: *Archaeometallurgy, Slag, Iron, Petrography, Elemental Analysis, Kuhdasht.*

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Introduction

Lorestan is bordered by the Eastern Zagros Mountains to the east, Kermanshah and Ilam Provinces to the west, and Khuzestan Province to the south. The existence of slag sites and their dispersion in different parts of Kuhdasht county as well as remnants obtained from industrial activities in the region, indicate the importance and attention of humans to metalworking activities in this region. The aim of this study was to investigate the existing environmental capability of the region for archaeometallurgy was done during three seasons of field surveys to 2014-2019 by the authors. Northern Kuhdasht county is located within the two valleys of Homian and Olad-Ghobad. This area is completely mountainous with deep valleys and covered with forests of oak. The distribution of sites indicates the widespread concentration of industrial activities in this part of the Iranian plateau. The identified sites have been formed uniformly on the slopes of the heights and the hills. In the survey study, slags are the most findings related to metalworking interactions which make it possible to identify the used metal, type of smelting and extraction ways.

Slag is a silicate debris from the melting process that appears as a molten silicate or a mixture of several silicates. Under certain conditions, slags can prove the presence of compounds such as oxides, sulfides, phosphates, borates, carbides, as well as metals in their natural form. Slags are classified according to their size, color, texture, degree of porosity, appearance structure, corrosion, and volumetric weight. In the metalworking industry, temperature control has always been considered and in the final stages, smelting has been done under oxidative conditions. Changes in the weight percentage of some materials in ancient sites determine the control of heat during melting.An Accessory substance added to the molten or fluxes is more from calcite and carbonate compounds which lower the melting point and thus provide the optimal use of the fuel. When higher temperatures are allowed in the furnaces, lime is added to the furnaces to increase the efficiency of the furnace during furnace loading. The melting temperature for slags with a high percentage of silica, iron oxide, and lime in an equilibrium state in their chemical texture is around 1100° C to 1200° C. In order to understand the method of metal smelting, the study of the structure and texture of the walls of the melting furnace is also of particular importance. In some non-ferrous slags that have high porosity, coal residues from annealing and melting operations can be observed. These carbonaceous compounds are very important for the chronology of slag using the C14 technique.

Description of slags discovered from Northern Kuhdasht county

All sites studied in this survey are the result of research in Northern Kuhdasht. This city is located in the west of Lorestan province (46°51’-47°50’E, 33°9’-33°56’N). there is a lot of evidence showing that the Central Zagros is one of the most advanced centers for metalworking in the Iron and Bronze Ages, but it is not clear when was the most usage of iron metal in this region. another question is whether the mineral resources and ores used for making objects in the region are local or imported; briefly, there are still many ambiguities about the bronze and iron period in the region (Fig. 1a, Fig. 1b).

Despite traditional archaeometallurgy in these sites, in terms of dating, the details of extraction method and the mines used, have not been addressed. Emami et al 2016, have been done a study in the area of Southern Kuhdasht county in Botkhaneh Cave, with samples of slag in relation to metal melting. They conducted lithological studies in the area and the identification of magnetite, hematite and pyrite veins was the result of this research.

Outcrops of chalcopyrite and yellow copper sulfides have been observed in some study parts

Fig. 1a: Geographical location of Kuhdasht in Lorestan province.

(Emami, et al. 2016). On the other hand, due to the dispersion and accumulation of surface slags, it cannot be acknowledged that in which part of the region, metalworking activities were carried out intensively or where is the possible location of the melting furnace? Only conduction of comprehensive field surveys and excavation of the mentioned sites, can lead to desirable results. In this research, physical and chemical examinations of slags in four sites and places related to metal melting interactions such as: Changari, Chalgheshaleh, Sargiz and Dalab are studied. In this research, the following questions will be answered. How was the metalworking system done in Kuhdasht region? What kind of furnaces was used to melting iron in the region? What materials have been used to help smelting in the furnaces of Kuhdasht region?

Due to the chronological complexities of the Kuhdasht region, a completely systematic sampling has been performed here, which has resulted in an accurate classification of the materials obtained in this region. The slags found can be generally classified into three categories:

1. Porous agglomerated slags: These slags are dark or black. Their porosity is either due to the presence of gas cavities created by the melting operation with heat and the release of volatile compounds, or in some cases, the presence of accessory minerals, has caused porosity in the slag texture. In many cases, charcoal compounds can be observed in the slag texture (Emami, 2004: 4).

2. Glass slags and conglomerate: These compounds often have been exposed to high temperatures (above 980° C), in which case either the molten glass silicate (SiO_2) has been cooled rapidly or many possible clay compounds in the furnace have been melted at high temperatures (Emami, 2004: 3).

3. Low-porosity compact and furnace bottom slags: In other words, the cooled debris of the

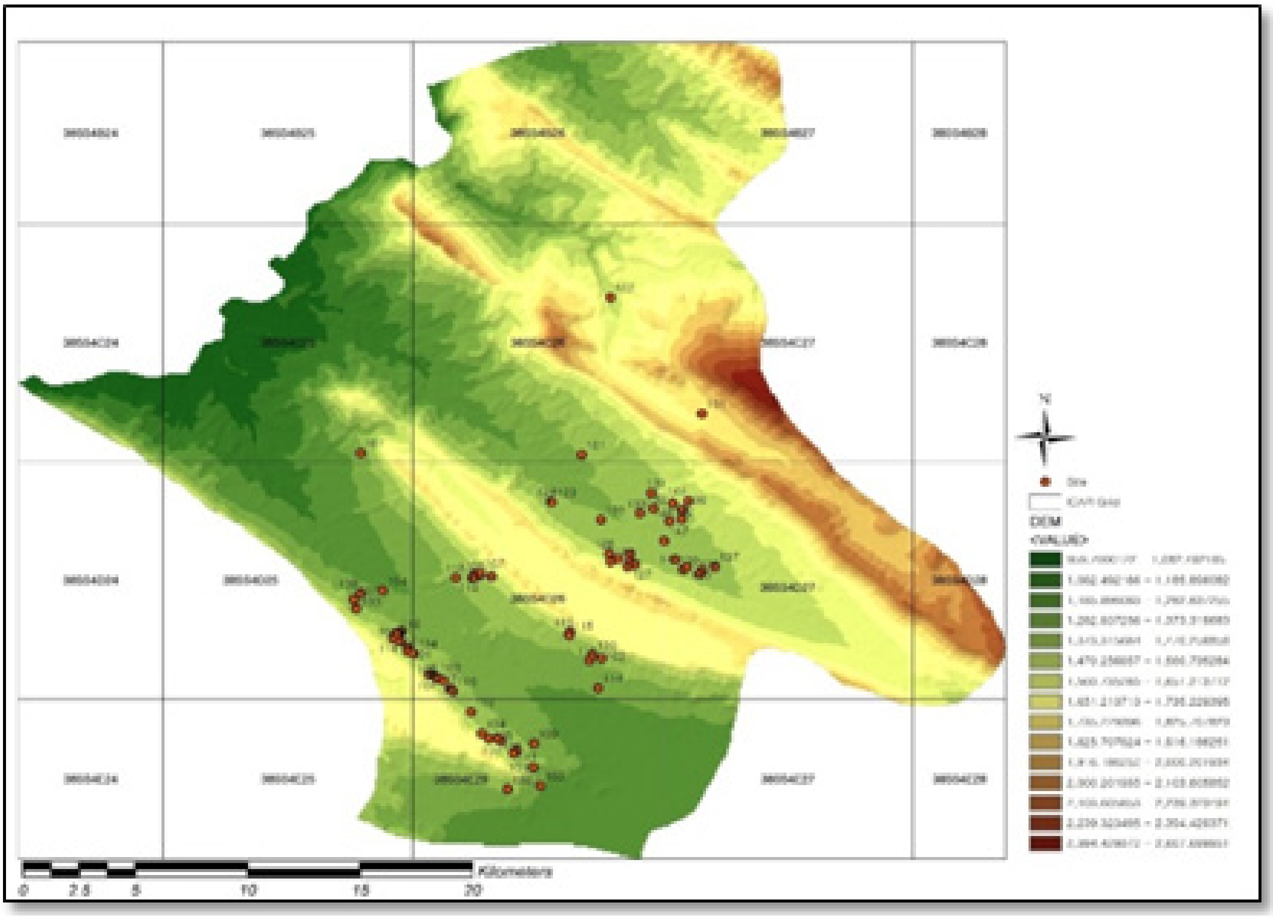

Fig. 1 b: Map of sites containe slag in North Kuhdasht county, Central of Kuhdasht city.

furnace floor, which with a certain curvature, has taken the shape of a semicircle of the furnace floor and in some cases, shows the effects of the presence of calcareous and carbonate compounds. The slag that usually forms at the floor of the furnace is called Kallote. What can be said about the archaeometallurgy industry in the study area; is the extensive use of iron. According to microscopic studies of thin sections the iron obtained in this area, are carbonate ore of iron and Volcano-Sediment iron ore.

Microscopic and chemical analysis of discovered slags in Northern Kuhdasht county

1. Results of Microscopic Studies of Slags

After field research and collection of slag samples and mineral stones from the sites, they were analyzed. the first part of the research was related to the study of their microscopic structure. The samples were analyzed based on mineral composition and differences in phase distribution. Microscopic studies were performed using binocular polarizing microscope in the thin sections (Table 1 and Fig. 2). Examination of the results shows that the main phase in most of them is Pyroxene and almost in all samples, metallic iron is observed. The tested samples were selected from four sites, namely: Changri, Chalgashalah, Sargiz and Dalab. The examination of their microscopic results is as follows: the five selected samples from the Sargiz site were significantly different under the microscope. SRGZ-8-3 is a porous sample, with glassy matrix which fine and acicular crystals of pyroxene has been formed inside the glassy texture (Fig. 2b). Its glass phase occurred due to the rapid cooling of the slag. Due to the high temperature, almost all of the mineral in it, is smelted and to a very small extent, vesicles of Iron Oxide are observed.

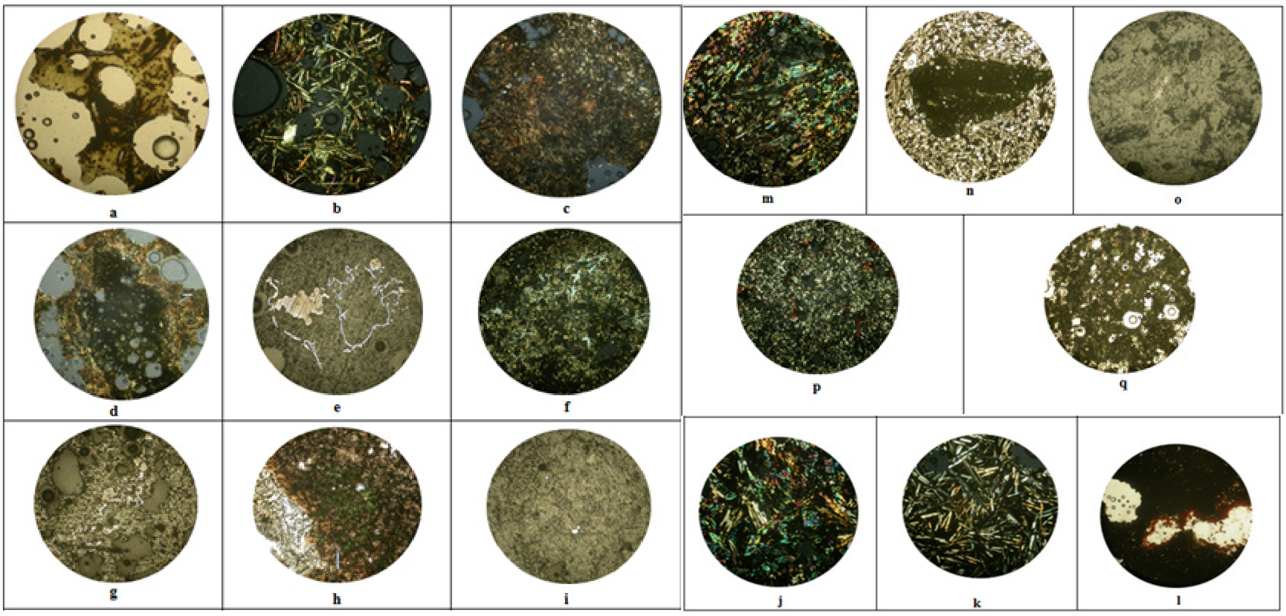

Fig. 2. Microscopic images of thin sections from a to q related to slag and ore samples from Northern Kuhdasht county by polarized binocular microscope.

Table 1. Results of Petrographic analysis of slag and ore samples in Northern Kuhdasht county

Metallic Mminerals	Silicate Mineral	Place	Sample Number
Metallic Iron vesicle	Pyroxene, Spinel or Glassy Matrix	Northern Kuhdasht/ Sargiz	SRGZ3-8-
Hematite, Metallic Iron	Melilite, Pyroxene, Spinel, Primary Mineral	Northern Kuhdasht/ Sargiz	SRGZ19-2-
Metallic Iron vesicle	Pyroxene, Spinel, Primary Mineral	Northern Kuhdasht/ Sargiz	SRGZ1-7-
Metallic Iron vesicle	Pyroxene, Melilite or Glassy Matrix	Northern Kuhdasht/ Sargiz	SRGZ2-7-
Metallic Iron vesicle, Hematite	Pyroxene or Glassy Matrix and Primary Mineral	Northern Kuhdasht/ Sargiz	SRGZ18-2-
Magnetite, Metallic Iron	Completely Dark Groundmass and Composed of Opaque Minerals	Northern Kuhdasht/ Dalab	DALAB1-
Wustite, Metallic Iron	Pyroxene, Spinel	Northern Kuhdasht/ Changari	CHEN5-
Wustite	Olivine, Spinel, with Spinifex Texture	Northern Kuhdasht/ Changari	CHEN2-6-
Metallic Iron	Melilite, Spinel, Pyroxene, with Glassy Matrix	Northern Kuhdasht/ Changari	CHEN1-3-
Magnetite, Hematite, metallic Iron	Completely Black Groundmass Composed of Opaque Minerals	Northern Kuhdasht/ Changari	CHEN3-6-
Wustite	Olivine, Pyroxene, with SpinifexTexture	Northern Kuhdasht/ Chalgashala	CHAL3-3-
Hematite	Melilite	Northern Kuhdasht/ Chalgashala	CHAL1-3-
Hematite, Metallic Iron	Olivine, Pyroxene, with Spinifex Texture	Northern Kuhdasht/ Chalgashala	CHAL4-6-
Hematite	Olivine, Pyroxene, Melilite with Spinifex Texture	Northern Kuhdasht/ Chalgashala	CHAL1-2-
Hematite, Metallic Iron	Pyroxene, Spinel	Northern Kuhdasht/ Chalgashala	CHAL2-6-

In samples SRGZ-2-19 and SRGZ-2-18, the Melilite mineral in microlithic form is abundantly seen in the matrix. Pyroxene mineral has been formed between the crystals of Melilite. It seems that due to the smelting of Iron in these samples and the lack of this element in the slag composition, the Olivine mineral could not be formed and instead, the abundance of Melilite mineral has been formed (Fig. 2b). Along with the Melilite and Pyroxene minerals, scattered Spinel crystals are also observed in the slag groundmass. Remains of smelted Metallic Iron vesicle and Hematite can also be seen in these two samples (Fig. 2 e). The SRGZ-7-1 sample is composed entirely of Pyroxene mineral. The Pyroxene mineral is found in two forms of swallowtail with dark green color, along with fine and light-colored Pyroxenes. Apart from Pyroxene, remnants of the mineral can also be seen under the microscope (Fig.s 2c and 2d). SRGZ-7-2 is also composed of fine-grained pyroxene mineral, along with Melilite star-shaped crystals in a glassy matrix (Fig. 2f).

One of the samples obtained in Dalab site is dark and entirely metallic mineral. These samples are dark in transmitted light and in reflective light composed of metallic iron oxide minerals (Fig. 2 g). Four samples have been selected from the Changari site; the CHEN-5 sample consisting of Pyroxene mineral with green Spinel mineral. In this example, the available metallic minerals are observed in the form of dendritic, which are composed of wustite mineral and Metallic Iron (Fig. 2 h, 2i). The CHEN-6-3 sample is dark in color due to the presence of large amounts of metallic minerals. The CHEN6-2 sample contains the Olivine mineral. This mineral is observed in skeletal and bladed form and shows Spinifex texture (Fig. 2 j). Olivine mineral is iron compound and fayalite type. In addition to Olivine in this sample, there is wustite which is seen as dendritic in reflected light. The CHEN-3-1 sample, similar to the Sargiz samples, is composed of the Melilite mineral, which is also accompanied by the Spinel and Pyroxene mineral (Fig. 2k). In this sample, the smelted mineral is iron, the evidence of which is well obvious in slags. CHEN-6-3 sample is dark in color due to the presence of large amounts of metallic minerals. The CHEN-6-2 sample contains the Olivine mineral. This mineral is observed in skeletal and bladed form and shows spinifex texture (Fig. 2 l). Olivine mineral is iron compound and fayalite type. In addition to Olivine, this sample contains the Wustite, which is seen as dendritic in reflected light. Five samples were studied from Chalghashala site. Three samples *(CHAL3-3, CHAL6-4, CHAL2-1)* have Spinifex texture. In these samples, Olivine mineral is abundant in dendritic and bladed form with Pyroxene, Spinel and Iron Oxide minerals (Fig. 2 l, 2m). Existing Wustite is seen as dendrites or in the form of vesicles and free iron droplets in the slag groundmass (Fig. 2 n). The CHAL-3-1 sample is composed entirely of the Melilite mineral, which is seen between the crystals of the Melilite mineral, the Hematite, with a dark groundmass (Fig. 2o). The CHAL 6-2 sample consists of dark metallic minerals, Pyroxene and Spinel minerals (Fig. 2 p).

2. Results of Chemical Analysis of Slags

There is no evidence regarding the identification of extractable mines in the region due to the lack of comprehensive field studies and lack of attention to industrial activities in this cultural zone , but geological studies in Lorestan province show that some areas Including the Northwest of Dorud, Khorramabad, the Mamulan in Pol-e Dokhtar, Mehrabkuh in Delfan, Haft Cheshmeh in Nourabad and the north of Koohdasht city in the area of Sorkh Dom Laki, have Iron veins and it seems Some of them have been considered in ancient times. Sorkh Dom Lori is located 10 km southeast of Kuhdasht village in Lorestan province, which was excavated in 1938 by the Holmes team and under the supervision of Eric Schmidt (Schmidt et al. 1989). Also, Sorkh Dom Laki area is one of the only identified areas in the north of the Lorestan that has been studied due to the existence of iron-bearing veins as well as ancient iron mines (Shishe-Gar, 2006: 304, 305). According to the type of metal and XRF chemical analysis performed on slags (Table 2),

Table 2. Chemical analysis of slags discovered in Northern Kuhdasht using XRF											
Sample%	Na2O	MgO	Al2O3	SiO2	P2O5	K2O	CaO	TiO2	Cr2O3	MnO	Fe2O3
SRGZ3-8-	0.098	2.7	18.8	33.2	1.1	0.21	16.7	0.39	0.036	0.11	26.3
SRGZ19-2-	0.076	2.2	22.3	34.9	0.61	0.22	15.4	0.33	0.030	0.14	23.3
SRGZ1-7-	0.11	2.8	18.9	34.4	0.53	0.21	14.1	0.34	0.05	0.13	28.1
SRGZ2-7-	0.07	2.7	17.8	37.3	0.69	0.41	25.6	0.38	0.05	0.16	14.5
SRGZ18-2-	0.12	2.6	20.5	33.11	0.33	0.21	18.2	0.48	0.031	0.16	23.9
DALAB1-	0.034	1.1	1.6	5.6	0.86	0.024	8.6	0.046	-	-	81.8
CHEN5-	0.067	3.1	14.9	26.1	0.47	0.19	13.4	0.25	0.029	0.32	40.8
CHEN2-6-	0.059	2.8	20.4	16.4	0.54	0.15	16.7	0.10	0.022	0.23	42.2
CHEN1-3-	0.098	2.4	26.4	34.1	0.63	0.24	15.9	0.36	0.060	0.18	19.3
CHEN3-6-	0.026	2.1	2.3	14.0	0.45	0.035	22.3	0.094	-	0.017	58.3
CHAL3-3-	0.11	9.4	11.4	27.9	0.58	0.11	28.3	0.33	0.012	0.12	21.5
CHAL1-3-	0.13	6.7	10.7	21.7	0.98	0.16	32.7	0.25	0.016	0.10	26.2
CHAL1-2-	0.13	4.7	13.1	28.9	0.49	0.17	20.7	0.36	0.024	0.15	31.0
CHAL2-6-	0.061	5.2	13.0	26.7	0.59	0.15	18.1	0.31	0.024	0.17	35.3

the mineral can be attributed to ore with a significant percentage of Manganese in some areas. Chemical analysis of the studied slags was performed using X-ray fluorescence spectroscopy, model 8420 from ARL company and UniQuant software for analysis of unknown samples in the Research center of conservation and restoration of cultural relics (Table 2).

In fact, mineral phases (Silicate and Metallic) are formed based on the ratio of Metal Oxides and Silica (SiO2). The results of chemical analysis show that the amount of SiO2 is between 5.6% - 37.3 the highest and lowest values of which can be seen in the samples related to Sargiz site. The amount of Fe2O3 is between 14.5% - 81.8 and Al2O3 is between 1.6% - 26.4 The highest amount of Fe2O3 is related to SRGZ-18-2 sample from slags in the form of various types of Iron Oxide, Hematite, Metallic Iron and Pyroxene. The percentage of MgO in the tested slags is between 1.1%-9.4. Increasing in the amount of MgO produces Olivine and Magnesium-rich Glass phase. CaO amount is between 8.6% and 32.7% and its increase also produces calcium-rich Olivine. The type of tested slag (Tap, Porous agglomerated, Low-porosity compact or furnace bottom slag) is highly influential in the results of chemical analysis and the Oxide ratio of the main elements (Metallic Oxides and Silica). In fact, the interpretation of chemical test data is based on the ratio of Metallic Oxides (FeO, MgO, MnO), Silica (SiO2), Aluminum Oxide (Al2O3) and Alkali oxides (CaO, Na2O K2O). The results of chemical analysis show that in the samples related to Sargiz, the amount of SiO2 and Al2O3 is very high in compare with the amount of Iron Oxide FeO. The increase in SiO2 and Al2O3 is a strong reason for the fact that the temperature has decreased during the melting operation. Also, high amount of CaO has provided the conditions for formation of Pyroxene Silicate phase. The results of petrographic microscopic studies also confirm that the main phase in Sargiz samples is Pyroxene. In SRGZ-2-19 and SRGZ-7-2 samples, Silicate Melilite Mineral has been observed. this mineral is formed when the percentage of SiO2 and CaO in the system is high, which is also confirmed by the results of chemical tests. In fact, this mineral is one of the characteristics of the melting system in the furnace, with the blowing process. In some cases, Melilite is formed alongside the Spinel, which is due to the equilibrium of the system, and in some cases alongside Pyroxene, which may have been due to reduction process.

According to petrographic results, Metallic Mineral in the form of vesicles or Metallic Iron droplets was observed in all samples in Sargiz. In the case of slags found in Chengari site, the results of chemical analysis show that on average the amount of FeO is higher than SiO2, which has led to the formation of Silicate Iron-bearing Melilite mineral, that is fayalite, in the CHEN-6-2 sample. In CHEN-3-1 sample, due to the high percentage of SiO2 and CaO, Melilite Mineral is observed along with Spinel and Pyroxene. Metallic mineral in these samples is mostly observed in the form of Metallic Iron droplets, Magnetite Fe3O4 and wustite FeO. In CHEN-6-3, Opaque minerals as well as most of Metallic minerals, have been seen. In the case of Chalghashlah slags, the percentage of FeO and SiO2 is close to each other, which has led to the formation of Silicate Olivine mineral in three samples of slags in this group. What can be said about the results of the analysis of this region; is the high amount of CaO that has caused the formation of the only silicate phase of Melilite in the CHAL-3-1 sample. Also, the percentage of MgO in the slags found in this site is high, which leads to the formation of Pyroxenes with high amount of Iron, Calcium and magnesium. In these samples, the Olivine and Pyroxene phases are the main Silicate phases, and the Metallic mineral is mostly seen as Metallic Iron and Hematite. Due to the amount of Silicate compounds, the amount of divalent iron (Fe3O4) increases when lime is added to the melting operation, so Silica stabilizes trivalent Hematite and lime stabilizes Iron Oxide as Spinel Magnetite. Only one sample from the Dalab site was examined. The results of chemical experiments show a high percentage of Iron Oxide Fe2O3 (81.8%) in this sample. Also, in the microscopic test of this sample, mainly the Metallic Minerals, Magnetite and Metallic Iron are seen. On the other hand, in the previous studies, the sponge texture was clearly visible in radiographic images, and this sample was detected as sponge iron.

Conclusion

By dating and laboratory studies, chemical and microscopic analysis, it can be concluded that the slags found in the metallurgy sites in the northern Kuhdasht in Lorestan, smelting activities and iron mining has continued from the Iron Age I (1500 BC) to the late Sassanid and the Islamic period. Also, in the general examination of the slags, the presence of sponge iron samples, parts of the furnace wall, slags around the blow pipe and the furnace floor slag, and the conformity of the visual observations with the laboratory results, all indicate that melting and smelting of iron metal in Northern Kuhdasht area has been conducted in air reduction furnaces and blowing air using blow pipes. Due to the changes in the percentage of CaO, it can be stated that this material has been added to the melting furnace materials during the melting operation to help the melting and lowering the temperature. also, the melting temperature for slags with a high percentage of Silica, Iron Oxide and Lime in equilibrium mode in their chemical texture was about 1100 ° C to 1200 ° C. In the microscopic results of some slags, primary minerals were observed, which are mostly in the form of Hematite and Magnetite metal minerals. This indicates that the used minerals are of magmatic origin, but to identify the sources of minerals used for melting and smelting of iron, it is necessary to study the reserves and resources available in the area and laboratory studies to analyze trace elements and isotopic.

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