# Iron Archaeometallurgy in the Triangle of the Sirdjān, Neiriz and Shahr- e- Bābak

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#### Abstract

During the course of our studies on archaeometallurgy in southeastern Iran, we tried to survey the triangle of the Sirdjān, Neiriz and Shahr- e- Bābak.

As a result, remains of old iron mining residues of smelting furnaces, and iron smelting slag heaps were observed and recorded. A few samples of iron ore and smelting slag have been analysed classically and instrumentally. According to the acquired results, it has been suggested that the blacksmiths of this region applied two methods to produce iron: first, producing sponge iron, and second, of molten cast iron. According to the classification and the dating, these metallurgical activities have been estimated as old as 500 years before the advent of Islam and that continued till the beginning of Islam.

Keywords: Iron Archaeometallurgy, Old Iron Mining, Iron Slag, Sponge Iron, Chāhak, Nārizi, Gol- e- Gohar, Molten Cast Iron

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#### Introduction

Iron is found in about 150 kinds of ores in the condition of porphyry and compound, however, the extraction of iron is economical from only 10 kinds of them. On the basis of the archaeological discoveries and the studies of ancient inscriptions, it was revealed that the Hittites, people who resided in northwest Iranian plateau and east of Anatoly, could smelted iron ore in 1300 B.C. (Gurney, 1992: 78). Archaeologists have also found iron objects in the Ur Royal Cemetery in Mesopotamia and Khofu Pyramid in Egypt which belongs to third millennium B.C. As there isnot any native iron, probably the origion of these iron had been aerolith. Anyway, investigations show that inhabitants in Tal- e-Iblis in Kermān province, Iran, had recognized iron in fourth millennium B.C. An iron pin among the metals objects belong to Iblis IV are very important in this respect (Pleiner, 1967: 400).

After this short introduction, let us now turn to some historical texts and reports. Studies show that there were mines and mining activities in various part of Iran such as Qazvin, Firuzkooh, Damāvand, Dāmghān, Semnān, Shāhrud, Kāshān, Gilān, Māzandarān, and some other sites (Zawush, 1996: 289- 300).

The clay Tablet 52 found in Persepolis is the oldest inscription that explains about iron workshops in this region. This tablet suggests

that Achaemenian arm makers were working in an area called Hankurraka under the supervision of an expert living in Nārizi. Nārizi is actually Neiriz located in Fars province in southern part of Persepolis (Cameron, 1948: 165). De Morgan, a French archaeologist, presents many useful informations about iron production in Gilān (probably Māsooleh), Māzandarān, west of Ouromiye as well as their export during Achaemenids (De Morgan, 1959: 9). Girshman writes that in spite of the development of metals utilization in this period, there was not any application for import. He thinks it happened because of the development of local industries (Ghirshman, 1987: 207) and Olmstead gives reasons such as, suitable economical techniques of smelting inside the territory, perfect security establishment, and insufficient transport expense for this matter (Olmstead, 1340).

In the third century of Hijri, Ebnu'l- Balkhi mentions iron mines located around a town named Ghatoreh which is probably Ghatrooeih (Ebne Balkhi, 1984: 128). This site is located on the main road of Neiriz- Sirjān. He, also, writes about other site named Chāhak, "... and iron and steel are extracted from that area (Sāheh) and changed in to blades called Chāhaky Swords ... " (Ibid: 125). Maghdasy reports on abundance of iron mines at Neiriz in the fourth of Hijiri (Maghdasy, 1982: 660). Estakhry and Ebne Hughel explain about iron mining around the proposed area under study

in the fourth of Hijira (Estakhry,1968; Ebne Hughel, 1966).

The writer of Nuzhat- al- Qulub reports on iron mines in various part of Iran such as Khāf, Ghohestān, Kaleybar in Azarbaydjān, and Kooreh in Tāremin of Qazvin meanwhile points the abundance of these mines at a site in Fars named Sāheh (Mustawfi of Qazvin, 1952: 660). He, that wrote his book at 740 Hijira, at other part of his itinerary reported on iron mine in Ghatoreh and Sāhak and explain about the latter is located in Fars province and that good steel is made of its iron (Mustawfi of Qazvin, 1919: 194).

Marcopolo, Venetian traveler, who visited Iran during Ilkhanid reports about iron mine of Koohbanān in Kermān and its iron smelting furnaces in his itinerary (Marcopolo, 1984: 47). Oleārius is the other traveler who visited Iran during Safavid. He explains about many things about this period, hance; reports about iron mining in Neiriz, Damāvand and Māsooleh (Oleārius, 1984). G. Zeydan has presented some explanation about iron mining in Fārs and Kermān (Zeydan, 1954: 112).

These are some examples of historical records pointing to ancient mining and archaeometallurgy activities in Fārs and Kermān. Unfortunately, in spite of the abundance of informations, fieldworks and surveys have rarely been done in this area.

Some archaeological surveys have so far been done in this area reveal remains of iron smelting furnaces and old mines (Stein, 1936: 206; Tylecote, 1970: 288- 89). The evidences implying iron mining and its smelting slags can be found from Abarghoo and Heneshk in Fārs upto Bāft and Djiroft in Kermān province (Wertime, 1967: 337). Evidences indicating iron archaeometallurgy have also been found in the north of Bāft, in Hazār mountains in Bideshk, Shooroieh, Zarangarān, and Piroodj (Pleiner, 1967: 389- 91). There are also reports on this type of mining and iron production using roasting during the 5<sup>th</sup> century of Hejira (Ibid: 379- 88).

There was old mining activities north of Neiriz, about 50 kilometer from Chāhak in Moshkān known as Kuh- e- Maadan. This type of mining has also been reported near Ghatrooeih (Tylecote, 1970: 288-9) which has been pointed by Ebn- e- Balkhi in 3<sup>th</sup> century of Hijira and by Mustaufi in 8<sup>th</sup> century of Hijira.

There are evidences indicating mining and iron smelting on the plains and valleys in the triangle of the Sirdjān, Neiriz and Shahr- e-Bābak. A large amount of ore has been extracted from the Gol- e- Gohar old mine located about 55 kilometers between Sirdjān and Shirāz in different times using either overground or underground methods. This old tunnel excavation has been carried out 120 meters long and 9 meters wide, and extracting

has been done 15 meters deep in a slope way (Fig. 1-3). Since there has been little smelting slag around this mine, it is accepted that ore had been carried to neighbouring area.

Iron smelting slags are scattered about 1 kilometer away from Gol- e- Gohar old mine. A lot of smelting slags can also be found 25 kilometers away from this mine in an area called Chāh Godār. Smelting slags are also seen in Beshneh and Yortanjir. These smelting slags together with pottery sherds are seen in a big land on the north of Neiriz in an area called Chāhak. The pottery sherds are parts of coneshaped dishes which are completely black and zinterred. Sir Aurel Stein, visited this area in 1936 and purported that it had been one of the most important centers of steel blades manufacturing (Stein, 1936: 209).

#### **Experiments**

#### 1. Samplings, analysis, and test instruments

The present research is a kind of historical, archaeological and experimental studies. Sampling was carried out statistically and specimens were analysed classically and instrumentally. Test instruments and analysis methods include:

- A. MAT- 625 Shtroline to measure Solphor.
- B. LAMBC A- 2 Perkin- Elmer spectrophotometer to guage Phosphorus.

- C. uv/vis (3100) Shimadzu spectrophotometer to measure Mn, Al, and Si.
- D. Varian Atomic Absorption to guage trace elements such as Mo, Cu, and Ni.
- E. Titration method was use to measure CaO and MgO.
- F. Wet method was applied to measure iron.
- G. The following instruments and methods were used to determine the age of two pottery sherds of Chāhak by thermolominesance methods:
- 7188 thermolominesance measuring instrument made by ELSEC, England.
- 7286 Alpha counting instrument made by ELSEC, England.
- Alpha Radiation Source (Am- 241).
- Beta Radiation Source (Sr 90- 40).

Measurements were carried out using computer code (7188), and age determination was done by personnel in charge of thermolominesance age determination of Iranian Cultural Heritage, Handicrafts and Tourism Organization and was controlled by computer code (TLD).

# 2. A proposal on iron smelting furnaces and result interpretation

Since iron has a high smelting point (1535° c) and it is difficult to be smelted, a lot of technological aspects should be taken into consideration to do the enterprise. That is why iron was known later than other metals.

However, to sort iron from ore, the experiences yielded from reduction and smelting of copper ore. But this was done gradually and with great effort because there are metallurgical and chemical differences between iron and copper (Rehder, 1991: 16). Archaeological evidences are indicative of accidental production of iron before the Iron Age (Wertime, 1967: 335). It is certain that man experienced iron extraction using lead and copper smelting furnaces and finally could produce sponge iron.

Sponge iron was also produced in the area under study like in many other ancient civilizations because the production of this type of iron has no need for technological knowledge comparing other forms of iron. In this method, ore had been grinded and, together with appropriate fuel, was being put into the furnaces which probably had the configuration of one of the two supposed designs (1, 2) (Fig. 4, 5). V. Pigott (1989: Pl. v) and N. Touhidy (1985: 98- 102) have presented similar to these type of supposed designs (Fig. 6- 8). Since charcoal has little smoke, high heat value, and easy combustibility, it was being used as an appropriate fuel in the archaeometallurgical activities (Horne, 1982: 6-13). Investigating the proposed area, scattered areas were seen with flora like wild pistachio and almond whose wood delivers high quality coal.

In the above mentioned furnaces, the openings in the wall stones are filled with clay

to avoid heat loss. Since blacksmiths had a lot of problems regarding inappropriate ventilation and discharging of supposed furnace no. 1(Fig. 4) after iron reduction, furnace no. 2 (Fig. 5) seems to be more efficient because enough oxygen is needed to reduce iron and effects the quality and the quantity of the activity.

Considering the furnace design and the raw material, the necessary temperature in these furnaces has been 1200°c to produce sponge iron. The extra gas of these furnaces together with unused air (full of Nitrogen) were sent out through the openings on the upper part of furnaces. To have such chimneys was very necessary because Co2 was to be evacuated and Oxygen replaced. Lack of such openings would cause the accumulation of Co2 in the furnace, and finally, the extinction of coal and the discontinuation of iron reduction (Greenwood & Earnshaw, 1985: 1244- 46). Since the blacksmiths had no idea to use the extra lime of the furnace, smelting slags were very sticky which caused the waste of so many iron.

Using lime in iron reduction brings up slag with fine properties which is easily sorted from iron. Good slag resulted from smelting should have 30% to 40% lime (Data Analysis Based on Information and Reports Presented by Esfahan Steel Plant). Experiments performed on the iron ore of this area show 0.2% to 7.5% lime (Table 1). The results of analysis done on the slags of the area also show a little lime: Chāhak slag 3%

to 11%, Gol- e- Gohar 1% to 15%, and Chāh Godar 1% to 10% (Table 2- 4).

Silica is also negligible although good slag is supposed to have 30% to 40% silica (Data Analysis Based on Information and Reports Presented Esfahan Steel Plant). by Experimental data show that Chāhak slag is from 1% to 7%, Gol- e- Gohar slag from 3% to 15%, and Chāh Godar slag is from 1% to 6% (Tables 2- 4). Since lime and silica have an important role in the reduction of smelting heat and the sorting of iron from slag, the shortage of these two elements in slag can be indicative of the high amount of iron, inappropriateness of slag (stickiness, high viscosity and density), and low quality of the produced sponge iron.

Lime and silica perform the following important reaction to produce slag (Stocchi, 1990: 427):

CaO+ Co2 
$$\triangle$$
 CaCo3

CaO+ SiO2  $\triangle$  CaSiO3 (Slag)

The slag is formed incompletely because of shortage of CaO and SiO2 in ore. Investigating the quality of slag and its analysis show pieces of coal between its layers and this fact is also indicative of high stickiness of slag and the storage of coal which has not been used because of lack of enough Oxygen.

According to the analysis of ore, the most important iron minerals in this area include Hematite (Fe2O3) and Magnetite (Fe2O4). The steps carried out in such furnaces are as follows: The initial process is the combination of Carbon and Oxygen and of Carbon and half of oxygen. Ore is reduced in the second step and sponge iron is produced. During the process, charcoal and Co are both reductants. Some Carbon is added to iron in the next step (Concidine and Concidine, 1984: 510- 20; Stocchi, 1990: 472).

The last step of reduction is along with the total consumption of coal. The furnace, having been cold, was destroyed, and produced sponge iron was taken out. Because of technical defects of these furnaces, the sponge iron contained impurities such as Silica, Phosphorus, Aluminium Oxide, Manganese Oxide, and other metallic Oxides whose existence in ore and slag have been proved by different experiments. Heating operation and long- run hammering were being done on the low quality sponge iron. In doing so, a lot of impurities were taken out of sponge iron and its Carbon was burnt and exhausted so that the iron could have more resistance (Buchner et.al., 1989: 372-4).

According to the evidences, a more developed technique in vogue in that area was the application of more complete furnaces whose production was molten cast iron rather than sponge iron. These furnaces used to apply more blowers and were charged with high quality coal comparing the amount of ore. It should be

mentioned that on the basis of some scientific suggestion the production of molten cast iron was possible in the past; for example, Chinese had molted iron and cast it like bronze in 700 and 600 B.C. (Rehder, 1991: 16).

The reaction of these furnaces were similar to the previous furnaces. The high availability of pottery dyes in Chāhak is indicative of applying the technique of cast iron smelting. These dyes are cone- shaped (fig. 9), and completely black and zinterred. Evidences referring to pouring molten cast iron in such dyes are quite obvious. Dissolving some of the material stuck to the walls and floor of these dyes into acqua regia adding some drops of Potassium and Thiocyanate to it showed the high concentration of iron. The quality analysis of this material also showed 40% to 45% iron. These dyes or crucibles were being used to mold the molten cast iron coming out of furnace. The coneshapedness of of these dyes caused the easy sorting of the slag from the molten cast iron so that slag would stay at the top and the cast iron at the bottom because of the difference of density (iron density is higher than slag density). Today, we use this property to design dyes for molding.

After cooling the molten cast iron, the mentioned dyes were broken and their content were taken out in the form of iron pieces or ingots. A brief investigation of other elements and composites in specimens is as follows:

- 1. A comparison among the amounts of Copper, Nickel, Molybdenum, and Manganese in ores and slags shows a shortage of such elements in the specimens of these areas (with the exception of Chāhak).
- 2. It is natural to see more Al2O3 in the slag than in the ore because this oxide is accumulated in the slag.
- 3. The amounts of the analysed specimens of Phosphorus and Sulphurous are very little. The scarcity of phosphorus is indicative of lack of apatite deposits in the area, and the shortage the Sulphurous shows the oxidization of ore in this area.
- 4. Analysis results reveal that the amount of MgO in the samples is less than the amount of lime. Thus, the limestone of this area is mainly common lime and isnot of the type of Dolomite.

#### 3. Oldness

The classification of the sherds collected from different area, specially in Chāhak, shows that the potteries belong to the historical periods, mainly Parthian and Sasanids, and specially to the beginning centuries of Islam.

The result of age determination of two samples of potteries of Chāhak shows that they are 1800± 200 and 1350± 100 years old. The results of this age determination are as follows:

- 1. Chāhak pottery sherds no. 1:
- The average of alpha particles counted per 1000 seconds: 8.66

-Potassium Oxide concentration (K2O): 1. 70%

-Thorium concentration (Th): 3.18 PPm.

-Uranium concentration (U): 4.64 PPm.

-Cosmic rays: 0.150 mGy

-Total errors: 11.69%

-Oldness: 1800± 200 years

2. Chāhak pottery sherds no. 2:

-The average of alpha particles counted per 1000 seconds: 7.71

-Potassium Oxide concentration (K2O): 1. 75%

-Thorium concentration (Th): 3.18 PPm.

-Uranium concentration (U): 4.64 PPm.

-Cosmic rays: 0.150 mGy

-Total errors: 8.03%

-Oldness: 1350± 100 years

#### Conclusion

Inscriptions and the written historical accounts show that archaeometallurgy in Kerman and Fars went on from the early time. Iron mining and archaeometallurgical operations were on of the most activities of man in this area. Field investigations and archaeological researches have confirmed these records. The present article, which is a combination of historical and archaeological investigations and laboratorial studies, showed that iron mining and smelting activities in the area under study continued

from the beginning of Ist century A.D. until the 10<sup>th</sup> centuries A.D. extensively. Research shows that more investigation of ancient mines and iron smelting centers will result in recognizing more mines and centers.

Sponge iron used to be produced in this area like other contemporary sites. The results of the analysis performed on the smelting slags and iron ores of this area are indicative of dearth of CaO and SiO2 which causes the waste of iron in slag and the increase of iron in it. Laboratorial studies on iron smelting slags and molding potteries which from Chāhak show that molten cast iron used to be produced in this ancient smelting center. The quality analysis of some of the material stuck to the walls and floor of these dyes showed the high concentration of iron. Blacksmiths had apparently applied more advanced furnaces which were equipped with different blowers to produce such type of iron.

On the basis of present investigation, probably blacksmiths of Chāhak had extracted iron ores from the mines of Moshkān, Heneshk, and Bāgh- e- Maadan and transfer them to this site. On the ancient route, leading to Beshneh and Yortanjir, we can find some iron ores that are similar to Gol- e- Gohar iron ore. So, in all probability, some iron ores have been smelted and restored by the side of the mines in which they extracted and sometimes they were carried to the other sites.

Table 1 Analysis of Gol- e- Gohar Iron Ore

Iron Ore	ppm	ppm	%	ppm	%	%	%	%	%	%	%
Sample of	Cu	Ni	P	Mo	SiO2	Al2O3	CaO	MgO	MnO	Fe	S
Gol- e-											
Gohar											
Area No. 1	21	50	0.02	4	trace	0.14	6.10	0.14	0.13	66.31	0.199
Area No. 2	73	74	0.01	12	3.10	0.13	2.09	0.17	0.12	68.24	0.152
Area No. 3	83	126	0.02	12	0.13	0.31	0.20	0.32	0.17	67.87	0.096
Area No. 4	138	127	0.10	8	2.05	0.16	1.38	0.17	0.06	69.91	0.094
Area No. 5	60	63	0.02	8	4.20	0.11	1.36	0.07	0.07	63.17	0.133
Area No. 6	51	70	0.06	n.d	Trace	0.16	0.69	0.21	0.09	67.35	0.054
Area No. 7	38	28	0.11	300	Trace	0.13	7.60	0.23	0.07	68.73	0.042
Area No. 8	238	76	0.08	4	0.78	0.31	0.65	0.18	0.17	71.72	0.230
Area No. 9	25	49	0.12	4	Trace	0.15	4.02	3.85	0.11	68.33	0.021
Area No. 10	240	350	0.07	20	6.80	0.07	7.40	0.90	0.09	68.46	0.076

n.d.: not detected

#### Iron Archaeometallurgy in the Triangle of the Sirdjān, Neiriz and Shahr- e- Bābak

Table 2 Analysis of Gol- e- Gohar Iron Smelting Slags

Iron Slag	ppm	ppm	%	ppm	%	%	%	%	%	%	%
Sample of	Cu	Ni	P	Mo	SiO2	Al2O3	CaO	MgO	MnO	Fe	S
Gol- e-											
Gohar											
Area No. 1	61	588	0.16	20	14.40	0.11	11.40	Trace	0.11	30.10	0.053
Area No. 2	63	126	0.29	20	5.19	0.71	11.80	1.48	0.09	46.73	0.090
Area No. 3	77	122	0.21	16	7.41	0.93	Trace	0.35	0.16	53.08	0.062
Area No. 4	72	134	0.21	16	3.01	0.52	4.89	0.64	0.19	38.28	0.071
Area No. 5	56	55	0.28	8	10.50	4.20	14.80	2.70	0.07	47.47	0.058
Area No. 6	55	63	0.01	n.d	3.14	Trace	13.30	0.48	0.18	52.83	0.166

Table 3 Analysis of Chāh Godar Iron Smelting Slags

Iron Slag	ppm	ppm	%	ppm	%	%	%	%	%	%	%
Sample of	Cu	Ni	P	Mo	SiO2	Al2O3	CaO	MgO	MnO	Fe	$\mathbf{S}$
Chāh Godar											
Area No. 1	51	42	0.42	n.d	3.62	0.26	9.30	2.70	0.14	50.57	0.041
Area No. 2	47	26	0.34	n.d	6.11	0.96	6.10	0.42	0.21	43.80	Trace
Area No. 3	66	50	0.30	n.d	3.50	1.05	8.91	4.60	0.13	48.65	0.035

Iron Ore	ppm	ppm	%	ppm	%	%	%	%	%	%	%
Sample of	Cu	Ni	P	Mo	SiO2	Al2O3	CaO	MgO	MnO	Fe	S
Chāhak											
Area No. 1	111	111	0.17	n.d	4.20	0.30	8.90	0.24	0.07	44.24	0.124
Area No. 2	32	n.d	0.14	n.d	1.80	0.35	10.21	0.22	8.28	36.53	0.025
Area No. 3	25	25	0.19	n.d	6.30	0.31	8.71	0.83	2.69	33.42	0.103
Area No. 4	84	164	0.22	n.d	4.19	0.64	10.70	0.20	0.13	50.56	0.149
Area No. 5	74	51	0.18	4	0.94	0.13	2.70	0.32	0.68	47.84	0.084

Table 4 Analysis of Chāhak Iron Smelting Slags

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## معدن کاری کهن آهن در مثلث سیرجان، نیریز و شهربابک

### رحمت عباس نژاد سرستی ا

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نگارنده، پیرو مطالعات و پژوهشهای جاری خود در خصوص فلزکاری باستان در منطقهٔ جنوب شرقی ایران به بررسی در بخش کوچکی از منطقه ای واقع در مثلث شهرستانهای سیرجان، نیریز و شهربابک مبادرت نمود.

در نتیجهٔ این فعالیت صحرایی و عملی، شواهدی از معدنکاری کهن آهن، بقایای کوره های احیا و محل های انباشت سرباره های ذوب آهن مشاهده، ثبت و ضبط شده است. تعدادی از نمونه های سنگ آهن و سربارهٔ ذوب که در محوطههای مورد مطالعه با روش آماری گردآوری شدند، با استفاده از روشهای کلاسیک و دستگاهی مورد آزمایش قرار گرفتند. بررسی نتایج حاصل از آزمایشهای شیمیایی انجام شده روی این نمونه ها نشان داده است که فلزگران این منطقه با استفاده از دو روش مختلف، موفق به تولید دو گونه آهن شدند؛ نوع اول، آهن اسفنجی و گونهٔ دوم چدن مذاب بوده است. بر اساس مطالعات انجام شده، قدمت فعالیت های فلزکاری موصوف به دوره ای که تقریباً از ۵۰۰ سال قبل از اسلام آغاز می شود تا قرون اولیه اسلامی مربوط می گردد.

واژگان کلیدی: فلزکاری کهن آهن، معدنکاری کهن آهن، سربارهٔ آهن، آهن اسفنجی، چاهک، ناری زی، گل گهر، چدن مذاب

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