Revista de Metalurgia
October-December 2023, 59 (4), e250
ISSN-L: 0034-8570
https://doi.org/10.3989/revmetalm.250

Characterization of iron nugget produced from iron ore concentrate in a microwave oven using a biomass-based reductant

Mustafa Boyrazli[a,*], Elif Aranci Öztürk[b], Yunus Emre Benkli[c], Canan Aksu Canbay[d]

[a] Firat University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Elazig, Turkiye

[b] Balikesir University, Balikesir Vocational School, Department of Machine and Metal Technologies, Balikesir, Turkiye

[c] Ataturk University, Faculty of Engineering, Department of Metallurgical and Materials Engineering, Erzurum, Turkiye

[d] Firat University, Faculty of Sciences and Humanities, Department of Physics, Elazig, Turkiye

(*Corresponding author: mboyrazli@firat.edu.tr)

Submitted: 15 March 2023; Accepted: 30 November 2023; Available On-line: 3 January 2024

ABSTRACT: This study is concerned with the characterization of iron nuggets obtained from the reduction of an iron oxide concentrate in a microwave oven using a biomass-based reducing agent. In the experiments, the concentrate of iron ore consisting of hematite and magnetite minerals supplied from Elazig region and containing 67.29% Fe after enrichment, and as a reducing agent, tea plant wastes containing 94.68% C and 0.03% S after carbonization was used. Carbon required for the reduction of iron oxides to iron was stoichiometrically added to the concentrate with a grain size of -45 μm after its basicity ratio was adjusted. The composite pellets produced after the addition of reducing agent and flux (CaO) were subjected to reduction in a household microwave oven at different times. After the process, optimum $(Fe_3O_4+Fe_2O_3)/C=1/4$, basicity ratio $(CaO+MgO)/(SiO_2+Al_2O_3)$ =1.2 results were obtained. It was seen that the metallic part contained 96.6% Fe, 2.4% C after being separated from the slag and that the metallic phase was separated from the slag very easily. As a result of the microstructure investigations, it was found that the product obtained had similar properties to white cast iron properties.

KEYWORDS: Biomass; Carbonized tea plant waste; Composite pellet; Iron nugget; Magnetite; Microwave reduction

Citation/Citar como: Boyrazli, M.; Ozturk Aranci, E.; Benkli, Y.E.; Aksu Canbay, C. (2023). "Characterization of iron nugget produced from iron ore concentrate in a microwave oven using a biomass-based reductant". *Rev. Metal.* 59(4): e250. https://doi.org/10.3989/revmetalm.250

RESUMEN: *Caracterización de pepitas de hierro obtenidas a partir de la reducción de un concentrado de óxido de hierro en un horno de microondas utilizando un reductor basado en biomasa.* Este estudio se ocupa de la caracterización de pepitas de hierro obtenidas a partir de la reducción de un concentrado de óxido de hierro en un horno de microondas utilizando un agente reductor basado en biomasa. En los experimentos se utilizó el concentrado de mineral de hierro compuesto por minerales de hematita y magnetita procedente de la región de Elazig y que

contenía un 67,29% de Fe tras su enriquecimiento, y como agente reductor, residuos vegetales de té que contenían un 94,68% de C y un 0,03% de S tras su carbonización. El carbono necesario para la reducción de los óxidos de hierro a hierro se añadió estequiométricamente al concentrado con un tamaño de grano de -45 µm tras ajustar su relación de basicidad. Los gránulos compuestos producidos tras la adición de agente reductor y fundente (CaO) se sometieron a reducción en un horno microondas doméstico a diferentes tiempos. Tras el proceso, se obtuvieron unos resultados óptimos (Fe_3O_4+Fe_2O_3)/C=1/4, relación de basicidad (CaO+MgO)/(SiO_2+Al_2O_3) =1,2. Se observó que la parte metálica contenía 96,6% de Fe, 2,4% de C después de separarse de la escoria y que la fase metálica se separaba de la escoria muy fácilmente. Como resultado de las investigaciones de microestructura, se comprobó que el producto obtenido tenía propiedades similares a las de la fundición blanca.

PALABRAS CLAVE: Biomasa; Magnetita; Pellets compuestos; Pepita de hierro; Reducción por microondas; Residuos carbonizados de plantas de té

ORCID ID: Mustafa Boyrazli (https://orcid.org/0000-0002-2340-6703); Elif Aranci Öztürk (https://orcid.org/0000-0001-8362-7332); Yunus Emre Benkli (https://orcid.org/0000-0001-7318-2552); Canan Aksu Canbay (https://orcid.org/0000-0002-5151-4576)

1. INTRODUCTION

Since the raw material and energy resources available in iron and steel production are becoming unable to meet the demands of the sector, studies in many countries focus more on the positive use of raw materials and energy resources and alternative new technologies to make production more economical. Sponge iron and iron ingot, which is a higher quality product than sponge iron, are some of the methods being investigated. These products can be used instead of scrap, especially in electric arc furnace facilities, or they can be used together with scrap (Çizmecioğlu and Sarıdede, 2005). Sponge iron, as an intermediate product, has introduced a different approach to iron and steel sector and overcome some negativities of scrap in terms of price and quality. However, sponge iron is produced at relatively low temperatures, and therefore, the gangue minerals in it cannot be completely removed, and hence, increase slag volume in subsequent smelting operations (Benkli, 2008). The ITmk3 process has attracted attention as an alternative method of iron and steel production in recent years. In this process, an ore or concentrate is mixed with a reducing agent and flux and a binder in a mixer, and then, is sent to the pelletizing disc or drums where self-reducing composite pellets are produced, which are then processed into iron nuggets between 1350 and 1450 °C in a rotary hearth furnace (Benkli, 2008; Kıkuchı *et al.,* 2010). Pig iron production using rotary hearth furnace technology is an alternate to blast furnace processes, and some other production techniques are also being investigated.

In conventional heating, heat is transferred to a material by conduction, convection and radiation. All the energy required to heat the material passes from the surface to the interior, and heat is limited by the flow rate, temperature and thermal diffusivity. Microwaves are electromagnetic waves with frequencies ranging from 300 MHz to 300 GHz. In a material that absorbs microwaves, heat can be produced inside the material, and the energy input accelerates the heating very quickly. Microwave heating reduces reaction activation energy, increases reaction rates and allows reactions at lower temperatures (Standish and Worner, 1990). Microwave applications allow reactors and production facilities to be reduced to smaller volumes and sizes for conventional heating (Sun *et al.*, 2005). Today, that kind of energy is used in such metallurgical processes as heating, drying, leaching, roasting, melting, carbothermic reduction of oxidized minerals and waste management (Clark and Sutton, 1996; Smith, 1993).

The term cast iron, identifies a large family of ferrous alloys. Cast irons are multicomponent ferrous alloys, which solidify with a eutectic. They contain major (iron, carbon, silicon), minor (<0.1%), and often alloying (>0.1%) elements. Cast iron has higher carbon and silicon contents than steel. Because of the higher carbon content, the structure of cast iron, as opposed to that of steel, exhibits a rich carbon phase (Doru and Stefanescu, 1990). Alloys with that carbon composition are completely liquid between about 1150 °C and 1425 °C. Cast irons solidify as heterogeneous alloys and always have more than one phase in their microstructure. During the solidification of iron- carbon alloys, the carbon is dispersed in austenite and eutectic cementite (Fe_3C) product or as a solid solution in graphite. Tea plant wastes used in this study are produced in thousands of tons every year in the plants producing black tea, especially in the Eastern Black Sea region of Turkey. Tea plant wastes are left to rot, and therefore, cause environmental problems although they have great potential (Güler *et al.*, 2017). Today, these wastes are used in fungi and ornamental plant cultivation (Gülser and Pekşen, 2003), briquetting (Demir, 2006), removal of heavy metal from wastewaters (Orhan and Büyükgüngör, 1993), pyrolysis into liquid and gas products (Tiftik, 2006), production of activated carbon/adsorbent (Gürten, 2008) and carbon nano tube synthesis (Güler *et al.*, 2017). So far, only the authors of this article have investigated the use of tea plant wastes as a reducing agent (Boyrazlı *et al.*, 2017).

One of the biggest problems of today is CO_2 emissions and greenhouse gases. It has been widely accepted that biochar is a high-quality fuel with high carbon, high calorific value and low pollution and can partially replace fossil fuels. Biochar has similar properties to raw biomass such as renewability, carbon neutrality, low sulphur content and low nitrogen content, which contribute to alleviating the fossil energy crisis and reducing CO_2, SO_2 and NO_x emissions. Biochar is generally porous and has high porosity, high pore volume and high specific surface area. In addition, the flammability and reactivity of biochar is better than coal. When it comes to iron production, if biochar can be used appropriately in the process, it can lead to energy savings, emission reduction, cost reduction, etc. targets can be achieved. Also, due to the purity of the biochar, high quality hot metal can be produced and provided with less impurities for the subsequent steelmaking process.

In this study, a plant-based product was used and it was also aimed to minimize CO_2 and greenhouse gases. This article is one part of study titled "Investigation of the Effect of Mechanical Activation on Microwave Reduction of Magnetite Ore Concentrate Using Carbonized Tea Plant Wastes." We have previously examined the microwave reduction of composite pellets with different stoichiometric and basicity ratios, and the effect of different particle sizes and mechanical activation on the reduction of iron oxides. This article provides information on the microstructure of iron grains after determining the optimal stoichiometric ratio, basicity ratio and oxygen gas amount. Not only did this study evaluate tea plant wastes but it also managed to produce a higher quality product than pig iron in a microwave oven in a shorter time.

2. MATERIALS AND METHODS

2.1. Materials

In the experiments, carbonized tea plant wastes including C and S content of 94.68% and 0.03%, respectively and a concentrate contained 67.29% Fe with a grain size of -45 μm and supplied from Elazığ region were used. The concentrate was composed of hematite and magnetite (70% Fe_3O_4, 30% Fe_2O_3) minerals. Calcined limestone (CaO) with a purity of 97.5% was used as flux to adjust basicity while producing cold-hardened composite pellets. Table 1 shows the chemical composition and Fig. 1 presents the XRD image of the concentrate used in the experiments.

2.2. Methods

The concentrate (30% hematite, 70% magnetite) with a grain size of -45 μm and carbonized tea plant wastes were mixed in the experiments such that $(Fe_3O_4+Fe_2O_3)/C=1/4$, $(CaO+MgO)/(SiO_2+Al_2O_3)=1,2$ in order to prepare composite pellets.

In the preparation of composite pellets, 10% of the materials (concentrate + flux + reducing agent) were taken from 30% molasses solution as binder, and 3% jelly was added to that solution. After the composite pellets produced were dried at 200 °C for 2 h in accordance with the production conditions of cold-hardened pellets in the literature (Bostancı *et al.*, 2017; Benkli *et al.*, 2018), it was observed that the strength values were 280 N/pellet on average and that it contained 20-24% porosity. These strength and porosity values are in accordance with cold-hardened pellet standards (Benkli *et al.*, 2018). It was determined that the molasses used as binders contained 33.12% C, 0.06% S, that its calorific power was 2428.3 kcal/kg, that the jelly contained 44.31% C and 0.03% S and that its calorific power was 3985.1 kcal/kg. The carbon content of the binders was also included in the stoichiometric calculations. Reduction was performed in a domestic microwave oven with a frequency of 2.45 GHz and 800 W. Experimental setup and crucible design are given in Fig. 2.

Table 1. Chemical composition of the concentrate

Component	Amount (%)	Component	Amount (%)
Fe	67.29	MgO	0.12
SiO_2	1.8	Na_2O	<0.01
Al_2O_3	0.26	MnO	0.09
$CaCO_3$	2.43	Cr_2O_3	0.116
K_2O	<0.01	P_2O_5	<0.01
TiO_2	<0.01	Pb	0.015
Ni	0.028	S	0.002
Ba	<0.005	Co	0.007
Zn	0.99	Cu	0.006
P	0.007	LOI	0.3

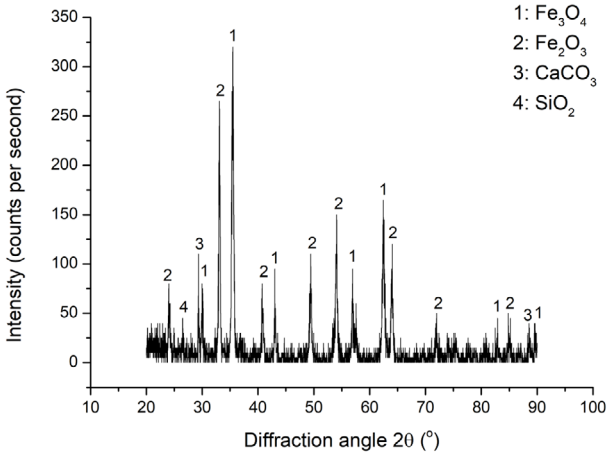

Figure 1. XRD image of the concentrate.

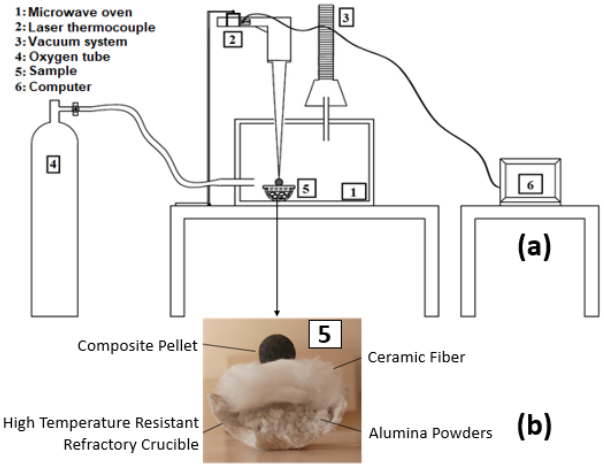

Figure 2. a. Experimental apparatus for reduction operations; and b. Design of crucible used in experiments.

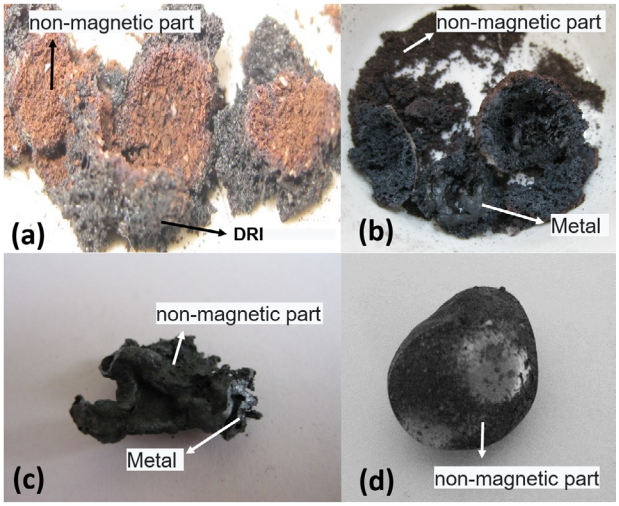

Figure 3. Macro image of pellet treated in microwave oven for; a. 5 min; b. 10 min; c. 15 min; d. 20 min.

It was seen that the CO-CO_2 gases released to the oven atmosphere as a result of the combustion of carbon in the composite pellet prevented the microwave-sample interaction and caused the reduction to stop in the preliminary experiments carried out in the microwave oven. Therefore, the ambient gases were vacuumed during the operation, and it was made sure that the microwave-sample interaction was at the same course. Moreover, the experiments were carried out by introducing oxygen gas at a velocity of 2 l/h to allow the carbon in the composite pellets to perform the Boudouard reaction and to give the required heat.

In the characterization studies of the obtained iron grain, macro-hardness measurement was performed using a DIGIROCK hardness tester, and micro-hardness measurements were performed in different parts of the sample using a Tronic, Digital Microhardness Tester HV-1000. Chemical composition was examined using a Foundry-Master metal spectrometer.

3. RESULTS AND DISCUSSION

Slag formation reactions and iron oxide reactions occurred for pure iron during the experiments performed on the composite pellets in the microwave. The macro images obtained as a result of treating the composite pellets in the microwave oven at different times are given in Fig. 3. It is seen in the image of the sample treated for 5 min that reduction was not completed, that the reductions of the samples treated for 10 min and 15 min were largely completed and melting occurred and that the sample treated for 20 min melted completely and that the slag phase was easily separated from the metal phase.

It is seen in the SEM image in Fig. 4b that there is no clear boundary between reduced and unreduced parts in the composite pellet consisting of concentrate, flux and carbonized biomass.

Since the concentrate consisting of hematite and magnetite minerals gradually fell from high-valent iron to low –valent iron in the experiments performed from 5 min to 20 min, silvery parts were observed to increase gradually in the SEM images (Fig. 4 (a - d)). When a certain degree of reduction was achieved, a porous non-reduced product formed in the shell form on the outside of the composite pellet while a completely reduced and a metallic core formed where melting occurred on the inside of the composite pellet. The unreacted shell almost completely disappeared due to the increase in treatment time (Fig. 3d).

The XRD patterns of the four samples removed from the oven after separation from the slag are shown in Fig. 5.

When XRD patterns are examined, it can be seen that the composite pellets consisted mainly of Fe_2O_3 and Fe_3O_4 before being reduced. The peak of Fe_2O_3 in XRD patterns disappeared after the 10-min treatment in the microwave oven, peaks representing FeO and Fe appeared. It was seen that all of Fe_2O_3 and most of Fe_3O_4 were converted to FeO and Fe after 15-min reduction. It is seen that FeO peaks in the 10- and 15-min samples disappeared in the XRD images of the pellet treated for 20 min, that the peak intensity of Fe gradually increased and that almost all of FeO was reduced to Fe. The CaO and SiO_2 peaks seen in the XRD peaks of the sample treated for 5 min means that the metal could not be fully separated from the slag. In the literature, such products are referred to as transition direct reduced iron (TDRI) (Arancı *et al.*, 2017; Bostancı *et al.*, 2017; Benkli *et al.*, 2018).

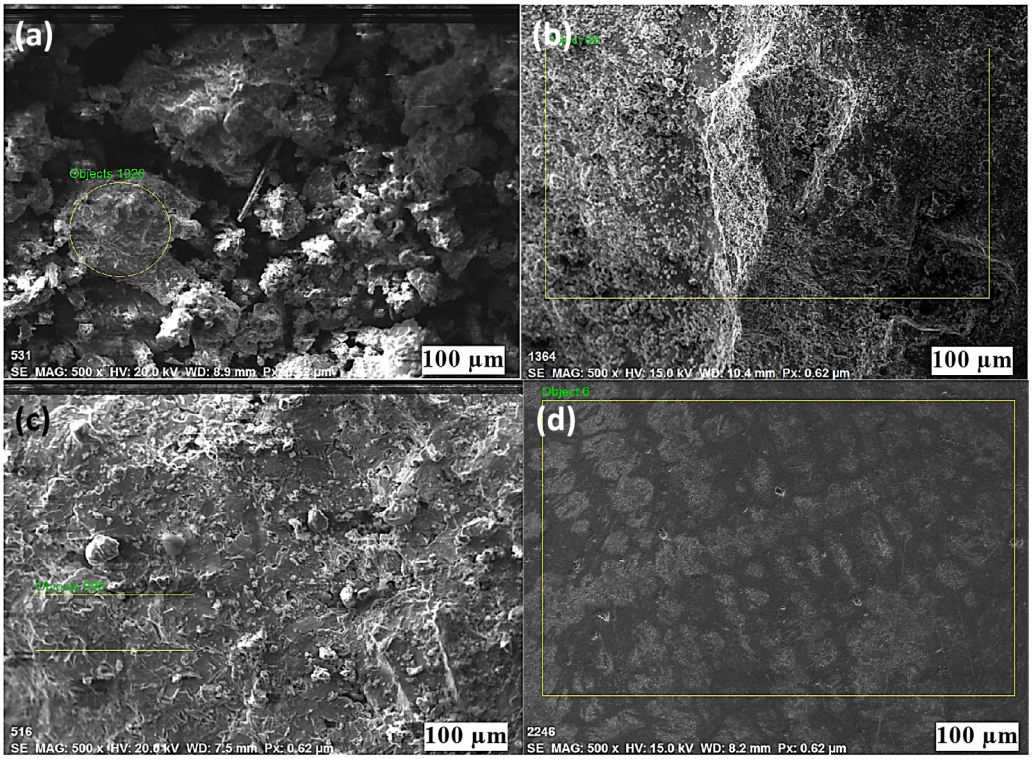

Figure 4. SEM image of pellet treated in microwave oven for a. 5 min; b. 10 min; c. 15 min; d. 20 min.

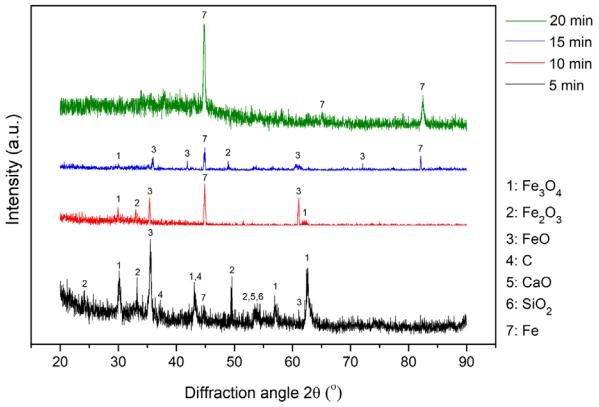

Figure 5. XRD patterns of microwave-treated pellets after separation from slag.

Microstructural examination is the most effective method used to identify cast irons, it is based on the form and shape in which the major part of carbon occurs with iron. The chemical composition ranges of unalloyed cast irons and blast furnace pig iron are given in Table 2.

In this section, the microstructure examinations of the sample, from which the most appropriate results were obtained as shown in Fig. 3d, were performed. Optical micrographs, XRD and SEM images were interpreted. In addition, EDX analysis, micro- and macro-hardness measurements, metal spectrometry and chemical composition determination of the sample were performed. According to the iron-carbon phase diagram, if sufficient carbon diffusion is achieved, iron nugget can be produced

Table 2. Chemical composition ranges for unalloyed cast irons and blast furnace pig iron (Peacey and Davenport, 1979; Smith, 1993; Agrawal *et al.*, 2000).

Element	Blast Furnace Pig Iron, wt.% *Peacey and Davenport, 1979*	White Cast Iron, wt.% *Smith, 1993*	Gray iron, wt.% *Smith, 1993*	Malleable iron (cast white), wt.% *Smith, 1993*	Ductile iron, wt.% *Agrawal et al., 2000*
C	4-5 (saturated)	1.8-3.6	2.5-4.0	2.00-2.60	3.0-4.0
Si	0.3-1	0.5-1.9	1.0-3.0	1.10-1.60	1.8-2.8
S	0.03	0.06-0.20	0.02-0.25	0.04-0.18	<0.03
P	<1	0.06-0.18	0.05-1.0	<0.18	<0.10
Mn	0.1-2.5	0.25-0.80	0.25-1.0	0.20-1.00	0.10-1.00

at furnace temperatures lower than 1400 °C. In that case, if the furnace temperature is between 1147 °C and 1400 °C, the austenite phase is formed after the reduction of magnetite to iron and carbon diffusion in solid state continues until the carbon content in the austenite phase is high enough in the melting zone (Kawatra *et al.*, 2005). The structure of the iron nugget obtained from the treatment of the pellet in the microwave oven in oxygen atmosphere for 20 min was examined and it was seen that it corresponded to the region referred to as white cast iron in the iron-carbon phase diagram. According to the iron-carbon phase diagram, white cast iron begins to solidify from its 100% liquid form (Kawatra *et al.*, 2005; Grote and Antonsson, 2008). When magnetite is reduced to iron, the carbon slowly dissolves in the metal, lowering the melting temperature of the iron and resulting in melting. The way and speed that that melt is cooled forms one of several cast iron structures.

After the produced iron nugget was taken into cold bakelite with polyester resin for metallographic examinations (Fig. 6), it was polished after sanding and then was etched with 4% Nital, and images were recorded at 1200x magnification on an optical microscope (Fig. 7).

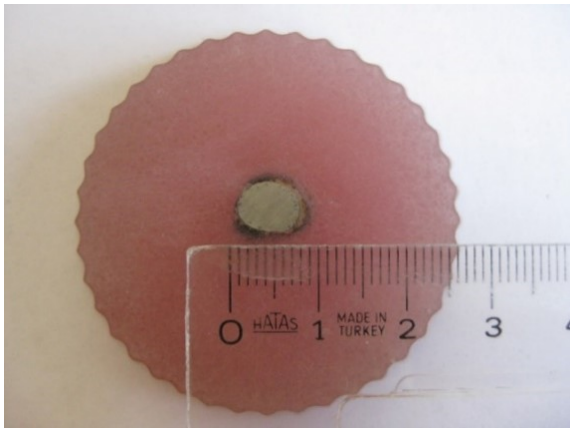

Figure 6. Cold bakelite of iron nugget.

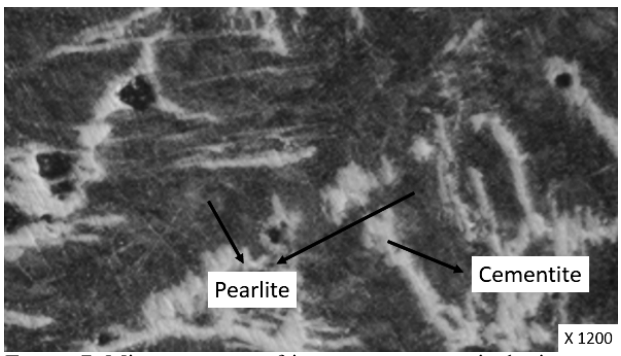

Figure 7. Microstructure of iron nugget on optical microscope after etching with 4% Nital.

The dark-coloured areas are pearlite while the reticulate white-coloured areas surrounding the pearlite areas are cementite. Iron nuggets were transformed into austenite dendrites while passing through the γ-Fe/liquid phase field during the first transformation, and then, this primary (eutectic) austenite was transformed into pearlite (lamellar iron carbide (Fe_3C) and α-ferrite layers). At 1147 °C, the remaining liquid solidifies to a eutectic morphology containing austenite (it cools down to pearlite) and iron carbide (Fe_3C).

A fingerprint-like image formed by different oriented pearlite structures, and surround this formation cementite, which is the hardest phase in the iron-carbon phase diagram, are seen in the SEM images in Fig. 8. In the larger magnification (Fig. 9), the lamellae forming the pearlite and the gaps between the lamellae and the ferrite by the solid state transformation of the austenite and the lamellar structure consisting of cementite layers (pearlite) are clearly seen.

The hardness values of the iron nugget obtained from the experimental studies were examined, macro hardness values are given in Table 3. The average of these results obtained as Rockwell hardness values is about 320 HVN. Micro-hardness measurements were performed in different regions of the same sample and were found to range from 288 to 904 HVN (Fig. 10).

The higher the amount of molten carbon (the amount of carbide) in the metallic structure, the higher the hardness. While an ordinary white cast iron alloy has a hardness of 374 to 649 HVN, they may contain primary iron carbides with a micro-hardness of 900 to 1200 HVN in a pearlitic structure with a micro-hardness of 220 to 300 HVN (Kawatra *et al.*, 2005; Grote and Antonsson, 2008). As a result of the microstructure investigations, it was concluded that the produced iron nugget had a white cast iron structure. White cast iron hardness increases with increasing carbon content depending on an increase in the amount of cementite (Fe_3C) and a decrease in distance between the lamellae of the pearlite (gap between Fe_3C and α-ferrite layers) with increased cooling speed. There are two types of iron carbides that contribute to the micro-hardness of the structure. The first type consists of carbides generated by solid-state reactions, the second type consists of carbides generated by liquid-state reactions. Diffusion of solid carbon is much slower than liquid diffusion. Thus, the large increase in hardness depends on greater and/or rapid diffusion of carbon in the metallized portion. In other words, the increased waiting time in the microwave oven resulted in an increase in the amount of eutectic cementite.

The XRD patterns of the product obtained in the microwave oven are given in Fig. 11. The carburi-

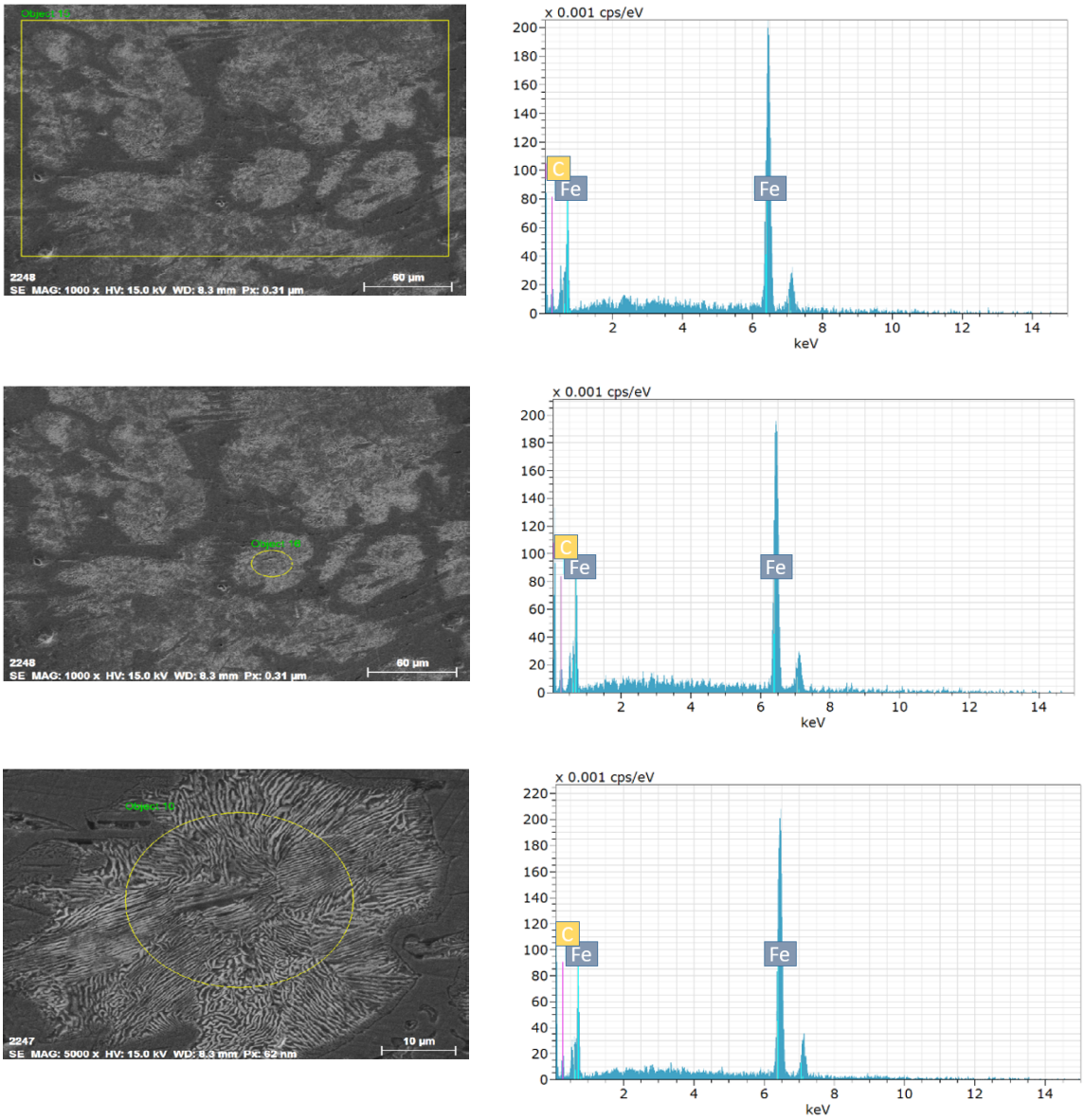

Figure 8. SEM images of iron nugget and EDX analysis from selected areas.

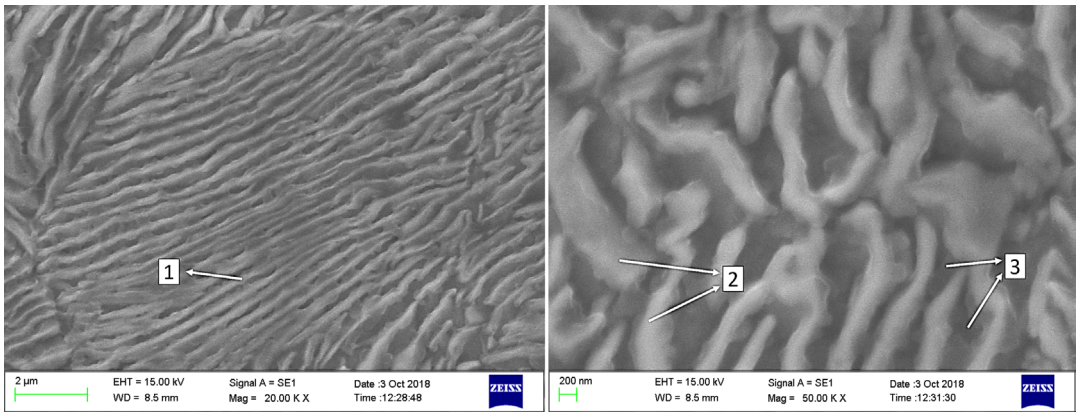

Figure 9. SEM microstructure images of the iron nugget. [1] Pearlite; [2] white lamellae are ferrite in lamellar structure; [3] dark areas between white lamellae are cementite.

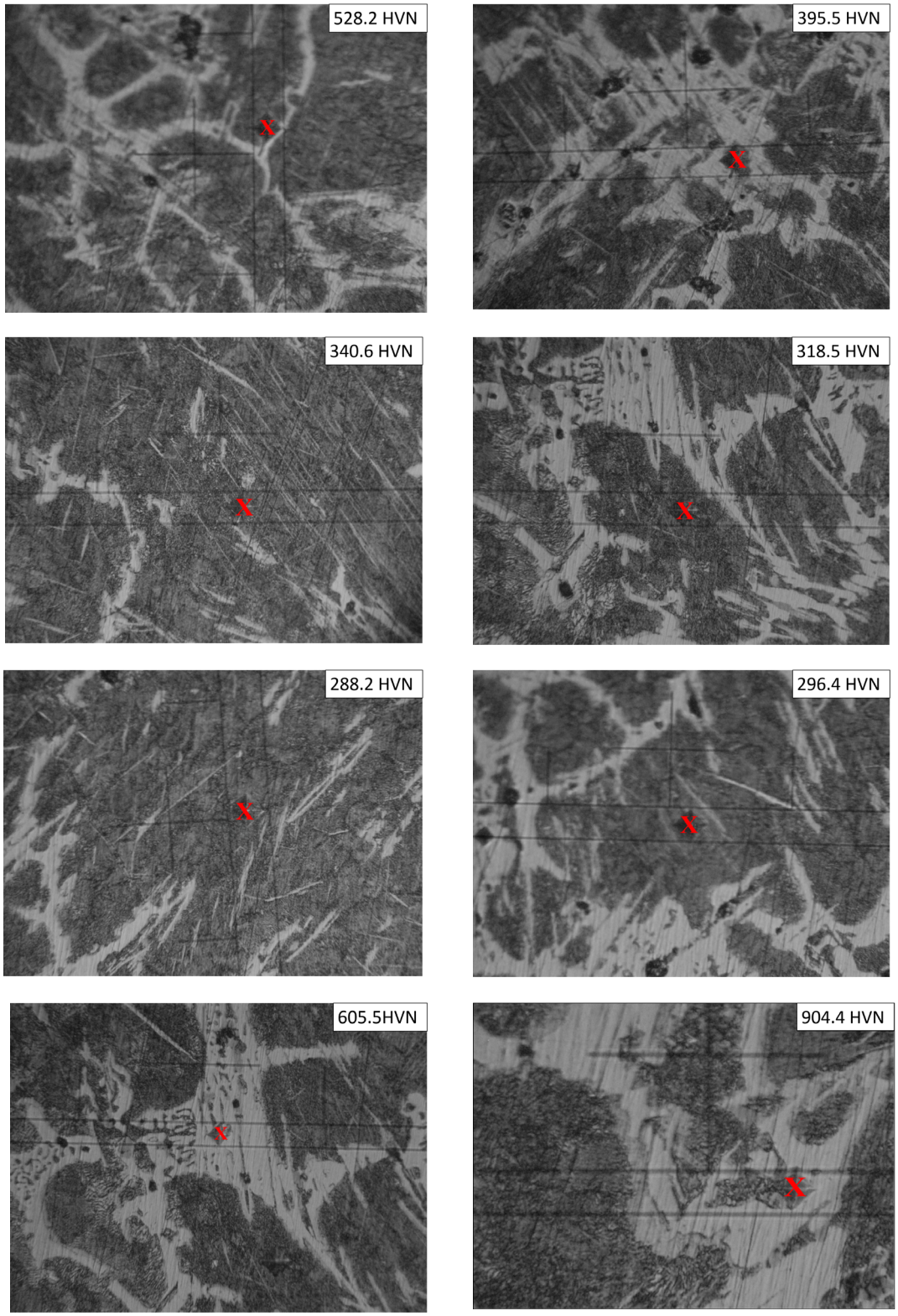

Figure 10. Micro-hardness values of phases indicated as "X".

Table 3. Macro hardness values of iron nugget

HRC
31.9
30.8
31.6
31.2
31.8

Average: 31.6 Standard Deviation: 2.8

zation and the liquefaction and aggregation of the metal resulted in the separation of the slag from the structure into a final product. Liquid carbon diffusion plays a key role in the production of a quality product similar to blast furnace pig iron. 20 min was found to be sufficient for that carbon diffusion to take place in the microwave oven.

The DRI-TDRI-Iron Nugget transition stage and the kinetics of transition reactions during the production of iron nugget samples are critical for the production of homogeneous pig iron nuggets of higher quality and with lower carbon content. However, during reduction production reaction of reducing gas (Boudouard reaction) and carburization and formation of wustite slow down reduction. The treatment of the iron nugget, which was thought to be obtained in a much shorter time in the microwave oven, increased to 20 min due to the slowness of those reactions. Slag formation reactions started on passing to the liquid phase, and the slag was found to be completely separated from the metal.

The chemical analysis of the produced iron nugget was performed using a Foundry-Master metal spectrometer. Table 4 shows both the analysis of the product obtained in this study and of those obtained under different conditions in the literature.

As it can be seen in Table 4, the iron grain produced in this study is of higher quality than the

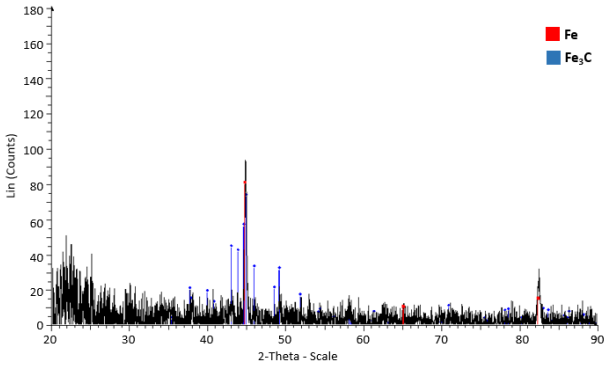

Figure 11. XRD results showing iron and cementite peaks in microstructure of iron nugget.

products and blast furnace pig iron produced in other studies. The fact that the carbonizing product of tea plant waste, a biomass, was used as reducing agent in this study resulted in a relatively low sulphur content. According to the chemical composition ranges for unalloyed cast iron and blast furnace pig iron in Table 2, the product obtained in this study is white cast iron. All carbon is compounded with cementite. All white cast irons are eutectic alloys and are obtained by rapid cooling from the solidification temperature. Its usual microstructure is made up of pearlite and cementite (Fe_3C). It is thought that the product containing 0.42% silicon was subjected to rapid cooling at the end of microwave oven treatment. When treatment starts in a microwave oven, the most heated zone shows the highest resistance to the microwave. When there is a change from solid to liquid in that zone, the microwave loses its effectiveness, and the local heating zone cools rapidly. In this study, the sample reached the liquefaction temperature, and the melting time was sufficient for the formation of metal and slag phases.

Table 4. The comparison of the product obtained in this study with those obtained under different conditions in the literature

Element	Blast Furnace Pig Iron, wt.% *Peacy ve Davenport, 1979*	White Cast Iron, wt.% *Smith, 1993*	Pig Iron Nuggets, wt.% *Kawatra et al, 2005*	Iron Nuggets, wt.% *Benkli et al, 2018*	Product obtained in this study, wt.%
Fe	90-95.5	93.3-97.3	95-97	94.9-95.4	96.6
C	4-5	1.8-3.6	1.7-3.5	3.6-3.8	2.4
S	0.03	0.06-0.2	0.2-0.8	0.6-0.5	0.14
P	<1	0.06-0.18	0.12	0.05	0.06
Mn	0.1-2.5	0.25-0.80	0.12	-	0.04
Si	-	-	0.8	-	0.42

4. CONCLUSIONS

In this study, the product obtained from the carbonization of tea plant waste was used as a reducing agent in a microwave oven. In this way, microstructure and characterization studies were carried out by producing iron nuggets from iron ore concentrate consisting of hematite and magnetite minerals. The results are as follows:

- The CO-CO_2 gases released to the atmosphere as a result of the combustion of carbon in the composite pellet prevented microwave-sample interaction and caused reduction to come to a halt in the preliminary experiments in the microwave oven.
- 20 min is a sufficient treatment duration for microwave production of a composite pellet of 70% magnetite and 30% hematite.
- The composite pellet, with a basicity ratio ($CaO+MgO/SiO_2+Al_2O_3$) of 1.2 and containing twice the stoichiometric ratio of carbon, was processed in a microwave oven in an oxygen gas atmosphere for 20 min. The resulting product has corresponded to the region called white cast iron in the iron-carbon phase diagram. According to the analysis performed using a metal spectrometer, the product contained 96.6% Fe, 2.4% C, 0.42% Si, 0.04% Mn, 0.14% S and 0.06% P.
- The optical micrographs revealed the zone of dark colored pearlite and the reticular white cementite zones surrounding the pearlite. It can be stated that the iron nugget was transformed into austenite dendrites while passing through the γ-Fe/liquid phase field during the first transformation, and then, this primary (eutectic) austenite was transformed into pearlite.
- The SEM images of the iron nugget presented a fingerprint-like image formed by different oriented pearlite structures and a cementite phase surrounding this formation. In larger magnification, the lamellae forming the pearlite and the gaps between the lamellae and the ferrite formed by the solid state transformation of the austenite and the lamellar structure consisting of cementite layers (pearlite) are clearly seen.
- It is emphasized in the literature that the production of iron grains with the desired microhardness depends on the processing time in the furnace (Benkli, 2008).
- As a result of the microstructure examinations, it was concluded that the product produced was white cast iron. The hardness of white cast iron increases as the carbon content increases, due to the increasing amount of cementite in the structure and the decrease in the distance between the lamellae of pearlite with increasing cooling rate. The large increase in hardness from DRI to TDRI and iron nugget is due to greater and/or faster diffusion of carbon in the metallized fraction. In other words, increasing the waiting time in the microwave oven caused an increase in the amount of eutectic cementite.

ACKNOWLEDGMENTS

This study was covered from "Investigating the Effects of the Mechanical Activation Process on the Microwave Reduction of Magnetite Ore Concentrates with Tea Plant Wastes" titled the PhD thesis.

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