

Investigation of Surface, Microstructural, Physical and Metallurgical Properties of Domestic and Imported Pellets

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Öz

Düşük kaliteli demir cevherleri (> %50 Fe içeriği) öncelikle yüksek fırında kullanılabilir hale getirmek için zenginleştirilir. Zenginleştirme sonrası en yaygın kullanılan aglomerasyon yöntemi peletlemedir. Bu çalışmada, üç farklı pelet türünün (biri yerli, ikisi ithal) yapısal, yüzey, fiziksel ve metalurjik özellikleri belirlenmiştir. Peletlerin yüzey morfolojileri, element ve bileşik analizi SEM, EDX, XRF ve XRD cihazları ile yapılmıştır. Peletlerin boyutu farklı boyutlardaki elekler kullanılarak belirlenmiştir. Peletlerin nem içeriği hesaplanmıştır. Peletlerin yük altında azalmasının belirlenmesi için RUL testi yapılmıştır. Peletlerin aşınma indeksi ve parçalanma indeksi tambur testi ile gözlemlenmiştir. Ayrıca, peletlerin serbest şişme indeksi ve azalma-parçalanma indeksi belirlenmiştir. Araştırmanın sonunda, Kardemir A.Ş. tarafından sağlanan üç farklı peletin özellikleri karşılaştırılmıştır. Ayrıca, peletlerin gözeneklilik miktarı ve faz dönüşümlerindeki farklılıkları incelemek için numunelere ısı işlem uygulanmıştır. Sonuçlar, yerli peletin ithal peletlere göre daha iyi kimyasal içeriğe sahip olduğunu ortaya koymuştur. Ancak, yerli peletin yüksek fırında ayrı olarak kullanılabilmesi için metalurjik özelliklerinin geliştirilmesi gerekmektedir.

Anahtar Kelimeler

“Demir Cevheri Pelet, Demir Oksit İndirgeme, Demir Cevheri Aglomerasyonu, Gözeneklilik, Yüksek Fırın, Şişme”

Abstract

Low-grade iron ores (> 50% Fe content) are primarily enriched in order to make these ores available at the blast furnace. The most widely used method of agglomeration after enrichment is pelletizing. In this study, structural, surface, physical and metallurgical properties of three different pellet types (one domestic and two imported) have been determined. The surface morphologies, element and compound analysis of pellets were performed with SEM, XRF and XRD devices. Size of pellets were determined by using different size of sieves. Moisture contents of pellets were calculated. RUL test was done for determination of reduction of pellets at under load. Abrasion index and disintegration index of pellets were observed with tumbler test. Furthermore, free swelling index and reduction-disintegration index of pellets were determined. At the end of the research, properties of three different pellets provided by Kardemir A.Ş. were compared. Also, heat treatment was applied to samples for investigating variance of porosity quantity and phase transforms of pellets. The results concluded that domestic pellet has better chemical content than imported pellets. However, metallurgical properties of domestic pellet should be developed to use domestic pellet in blast furnace separately.

Key Words

“Iron Ore Pellet, Iron Oxide Reduction, Iron Ore Agglomeration, Porosity, Blast Furnace, Swelling”

1. Introduction

The development and maintenance of a vibrant national economy necessitates securing an appropriate position within the international economic system, as this is a means of raising the living standards of the people and ensuring the country's long-term development. Therefore, the priority of the domestic market is the advancement of its core sector, industry. Iron metallurgy encompasses many industries, the most notable of which are iron and manganese mining and enrichment, non-metallic raw materials, smelting of cast iron and steel, processing of metal products, production of electrical alloys, coke and refractory material production; recycling of metals is also included in this scope (Mourão, 2022). The iron and steel sector is of vital importance to Turkey's economy. Steel production is

also the highest consumer of electricity and fossil fuels, accounting for more than 25% of hazardous atmospheric emissions from the country's industrial sources and more than 30% of carbon emissions. In sustainable industrial development, it is important to consider socio-economic, technological, and environmental factors, mineral resources and fossil fuels, production facilities, steel demand, and export structures. In addition, energy efficiency, raw material quality, equipment and technology status, environmental impact, capacity utilization, dependence on exports, and other related challenges should also be examined in detail (Derkach et al., 2020).

Iron ore agglomeration technology has developed rapidly thanks to advances in the iron and steel industry. After World War II, sintering and pelletizing processes gained greater importance in parallel with the iron and steel industry. In particular, pelletizing technology was developed due to the depletion of high-grade ore reserves (Pimenta & Seshadri, 2002). Pelletizing plants were established to utilize low-grade ore reserves. Thus, these plants have positively impacted the iron and steel industry (Yıldız, 1990). Since the 1950s, pelletizing plants have been producing pellets for blast furnaces (BF) and direct reduction (DR) (Mourão, 2008; Narçin et al., 1995). Over time, two other processes have been developed that offer greater capacity, improved efficiency, and higher-quality pellet production (Shatokha, 2016). Over the years, lump ore has been the main iron material in blast furnace charges. With the depletion of rich and hard ore reserves, agglomeration technologies (sintering and pelletizing) have been developed to agglomerate the increasing amounts of ore dust and concentrates produced in mining. Thus, in addition to lump ore, sinter and pellets can also be used. Sintering technology uses coarser iron ore fines (-6.3 mm / +0.15 mm), while pelletizing processes concentrates and ultra-fine ores (-0.15 mm). When analyzing global iron ore reserves and deposits, it is clear that iron content is declining and that more intensive concentration steps will be necessary in the future to adapt to this. As ore processing intensity increases, so does the production of ultra-fine particles. Pelletizing is the only technology that can be used to efficiently agglomerate these ultra-fine ores and iron concentrates. In this scenario, iron ore pelletizing is growing and this trend will continue. The global capacity, which was 350 Mt in 2000, has now exceeded 600 Mt in 2020 (Kordzadeh et al., 2017).

During iron oxide reduction, changes in structure, sintering of oxides/iron, and changes in oxygen concentration in the lattice structure cause volume changes. It has been found that such volume changes cause the fragmentation of iron ore pellets and irregularities in the furnace during reduction. Numerous studies have been conducted on various iron ore pellets, and the growth of fibrous iron during reduction has been identified as the main cause of pellet swelling and strength loss. It has been found that reduction parameters, in addition to chemical composition and pelletizing parameters, also affect the growth of iron whiskers during reduction. Among these parameters, ignition temperature, ignition time, pellet porosity, reduction temperature, reduction gases, partial pressure, and flow rate have been reported to affect the pellet swelling index (Sharma & Prakash, 1992; Shigaki et al., 1986; Sharma, 1994; Al-Kahtany & Rao, 1980). Raw materials are important in blast furnace technology in terms of production speed and product quality. Therefore, the physical, chemical, and metallurgical properties of raw materials are examined before being used in the blast furnace (Dey et al., 1993). However, there is no global standard for evaluating the properties of iron ores. Local standards are used in countries to evaluate the properties of iron ores (Harrison, 1971).

The ore containing iron oxide that enters the blast furnace decomposes into oxygen and iron at high temperatures, with the oxygen combining with carbon to form CO and CO₂, which rise within the blast furnace. The iron, meanwhile, collects at the bottom in a molten state. As the air supplied to the furnace from the bottom and the gases decomposed inside the furnace rise upward, the reaction changes, causing the reduced ore inside the furnace to melt very rapidly and in large droplets. In the development of the blast furnace process, product speed, product quality and economy, production efficiency, and the raw materials used are very important in this operation. The suitability of iron ore for use in blast furnaces is determined by the physical and chemical properties of the ores. In accordance with the quality standards applicable to iron ore everywhere, local standards, particle size, moisture content, reduction under load, fragmentation indices (under load or at high temperature), free expansion index, and chemical content, etc., are taken into account regarding the quality and effects of iron ores. The aim of this study is to investigate and compare the structural, surface, physical, and metallurgical properties of three different pellets (two imported pellets and one domestic pellet) used in the blast furnace process at Kardemir A.Ş. according to Kardemir A.Ş. pellet specifications.

2. Experimental Procedure

2.1. Pellet Materials and Heat Treatment

In this study, three different pellets which are used in Kardemir A.S. were investigated. One of the pellets is domestic, others are imported which were called east pellet and ferrexpo pellet. Production of these pellets were occurred in Sivas/Turkey, Helsinki/Finland and Poltava/Ukraine, respectively. Heat treatment was applied to samples for investigating variance of porosity quantity and phase transforms of pellets. For this process, samples were heated to 950 °C and hold at this temperature during 3 hours. After that, samples were cooled slowly at furnace.

2.2. Surface Morphologies, Compositional and Crystal Structure Analyses of Pellets

The morphologies of the pellets were examined using a Zeiss EVO MA10 scanning electron microscope (SEM) at 10.00 kV, with magnifications ranging from 0.25 to 5.00 KX. Their compositions were analyzed using EDX. X-ray diffraction machine (Rigaku Ultima IV) with fixed monochromator was used in the range of 0-90° and X-ray Device was operated at 40 kV and 40 mA during measurements. For XRF analysis, samples were pulverized with using Siebtechnik GmbH machine. After this process, pulverized pellets were formed to tablet with fusion process. ARL Advant'x X-Ray Fluorescence Spectrometer was used to XRF analysis.

2.3. Analysis Used For Physical Properties of Pellets

For sieve analysis, sieve analysis test equipment (UTEST UTG-0420) were used. Size of pellets were classified according to limits: +16 mm, 16-12.5 mm, 12.5-10 mm, 10-6.3 mm, -6.3mm. Samples were heated to $105 \text{ }^\circ\text{C} \pm 5 \text{ }^\circ\text{C}$ in drying oven (İrmeco SLW100STD). Weight of samples were measured before and after this process. Weight of moisture and other components were calculated. Tumbler test was done according to ISO 3271 test standard. Samples were prepared (+6.35 mm in sizes and 11.3 kg in weight) for this test. Prepared pellets were fed to drum which has 1000 mm diameter and 500 mm width. After feeding, pellets were rotated at 25 rpm during 8 minutes. After this process, abrasion index and disintegration index of pellets were determined.

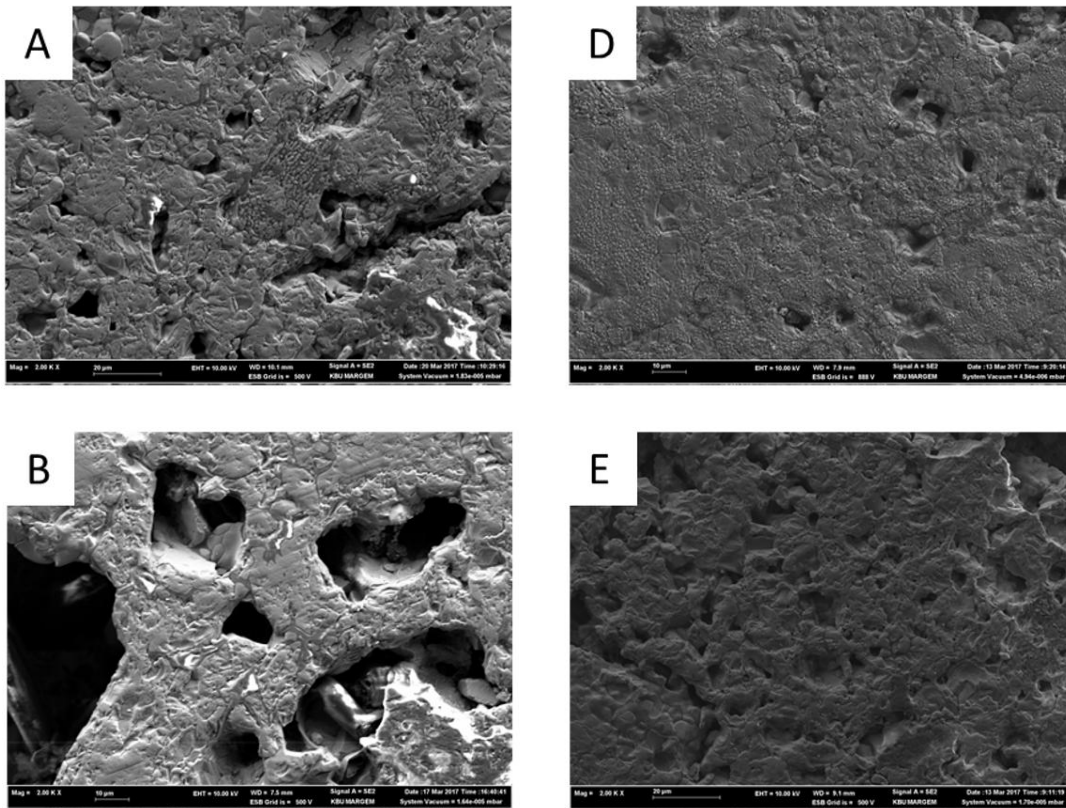
2.4. Analysis Used For Metallurgical Properties of Pellets

RUL (Reduction Under Load) test was done according to ISO 7992 standard with using R.B. Automazione test system for determination of reduction of pellets at under load. In this test, samples were heated to $1050 \pm 10 \text{ }^\circ\text{C}$. After starting of reaction, mix gas ($\%40 \text{ CO} + \%2 \text{ H}_2 + \%58 \text{ N}_2$) was fed to system and 50 kPa load was applied to sample during test. LTD (Low Temperature Reduction-Disintegration) test was done according to ISO 13930 standard with using R.B. Automazione test system for determination of low temperature reduction-disintegration index of pellets. In this test, pellets were fed to rotating tube with gas ($\%20 \text{ CO} + \%20 \text{ CO}_2 + \%2 \text{ H}_2 + \%58 \text{ N}_2$) at $500 \pm 5 \text{ }^\circ\text{C}$. Pellets were rotated at 10 rpm during 60 minutes. R.B. Automazione test system was used for investigation of FSI (Free Swelling Index) of samples. Test was done according to ISO 13930. In this test, 18 pellets were heated to $900 \pm 10 \text{ }^\circ\text{C}$ while N_2 gas was fed to system. After system temperature was reached to $900 \text{ }^\circ\text{C}$, mix gas ($\%30 \text{ CO} + \%70 \text{ N}_2$) was fed to system instead of N_2 gas. Reduction of pellets were continued during 60 minutes.

3. Results and Discussions

3.1. Surface morphologies, compositional and crystal structures of Pellets

SEM images of heat-free east, ferrexpo, domestic pellets and heat treatment east, ferrexpo, domestic pellets are shown in Figure . Porosities of pellets were reduced after heat treatment as seen in Figure . EDX composition analyses of heat-treated pellets are shown in Figure 2, and iron ores have uniform dispersions in all pellet types.



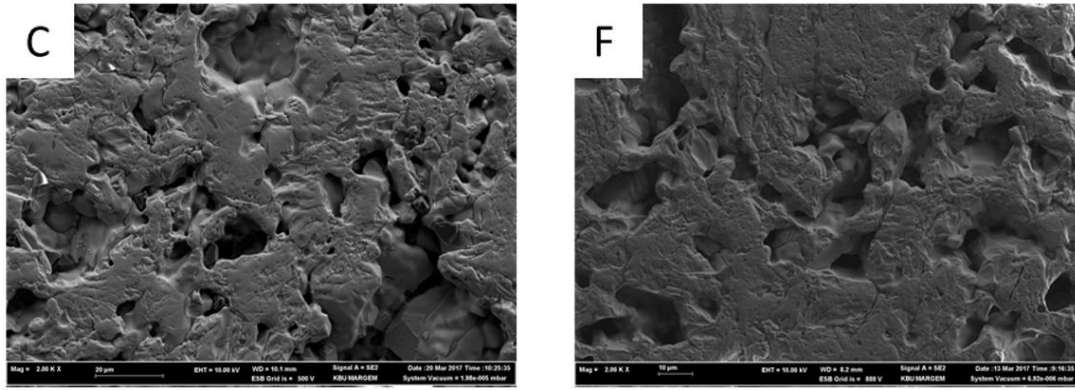
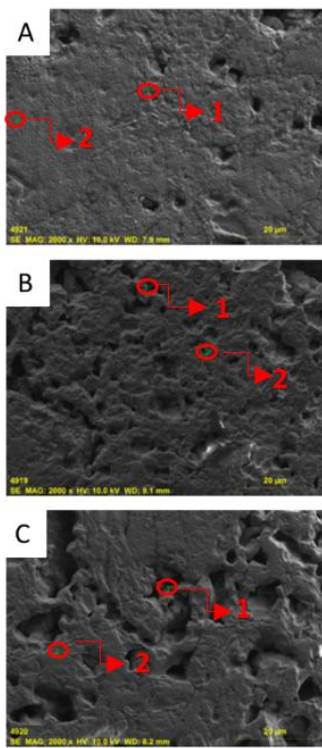


Figure 1. SEM images of a) heat free east pellet, b) heat free ferrexpo pellet, c) heat free domestic pellet, d) heat treatment east pellet, e) heat treatment ferrexpo pellet, f) heat treatment domestic pellet.



Atomic percent (%)										
	C	O	Na	Mg	Al	Si	K	Ca	Mn	Fe
1	2.20	72.41	0.27	0.87	0.65	0.11	0.06	0.13	0.00	23.29
2	1.77	68.28	0.25	0.91	0.66	0.10	0.04	0.00	0.26	27.71

Atomic percent (%)												
	C	N	O	K	Na	Mg	Al	Si	S	Ca	Fe	Zn
1	3.27	2.37	77.40	0.01	0.34	0.35	0.28	0.81	0.01	0.31	14.68	0.18
2	2.53	1.80	74.94	0.08	0.19	0.19	0.26	0.27	0.02	0.05	19.40	0.08

Atomic percent (%)										
	C	O	Na	Mg	Al	Si	P	Ca	Fe	Zn
1	3.87	83.96	0.03	0.44	0.47	0.30	0.04	0.04	10.27	0.59
2	2.27	87.92	0.22	0.41	0.43	0.47	0.08	0.00	7.98	0.20

Figure 2. SEM-EDX Analysis A) Heat treated east pellet, B) Heat treated ferrexpo pellet C) Heat treated domestic pellet.

The XRF analysis results for pellets are shown in Table 1. According to the analysis results, the highest iron content was observed in the domestic pellet.

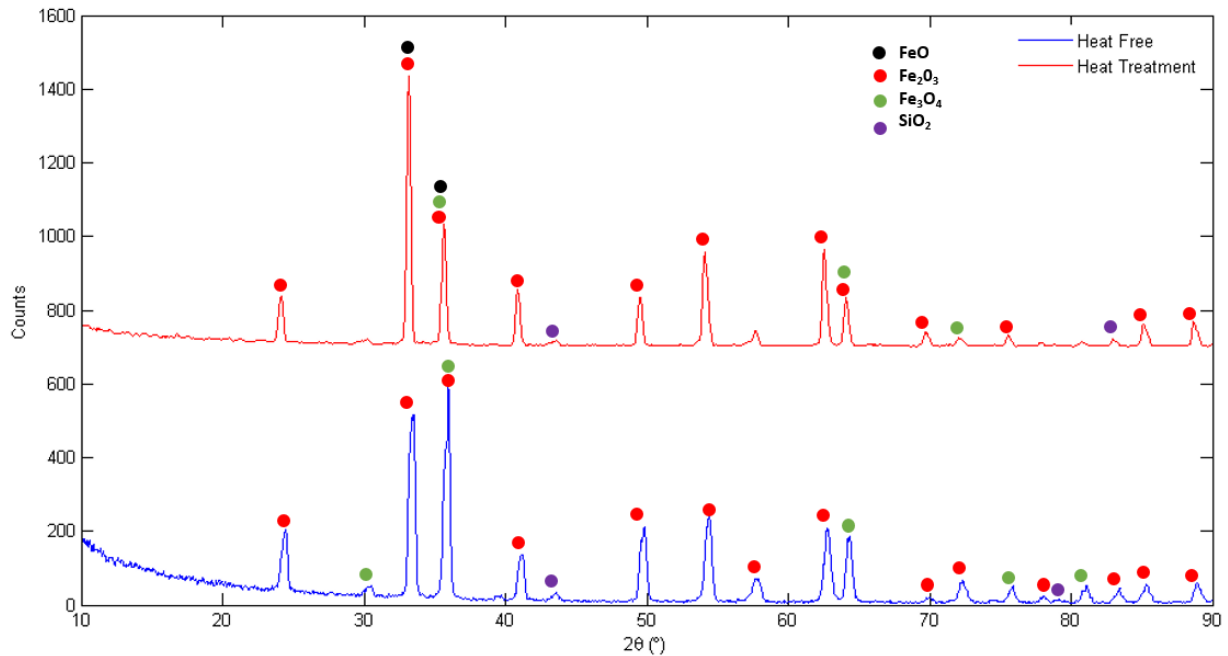
Table 1. XRF analysis results of Pellets.

%	Ferrexpo Pellet Sample	East Pellet Sample	Domestic Pellet Sample
Fe	65.02	60.78	66.23
SiO ₂	5.04	3.63	2.24
CaO	0.59	1.26	0.64
Al ₂ O ₃	0.55	2.60	0.78
MgO	0.36	2.02	1.05

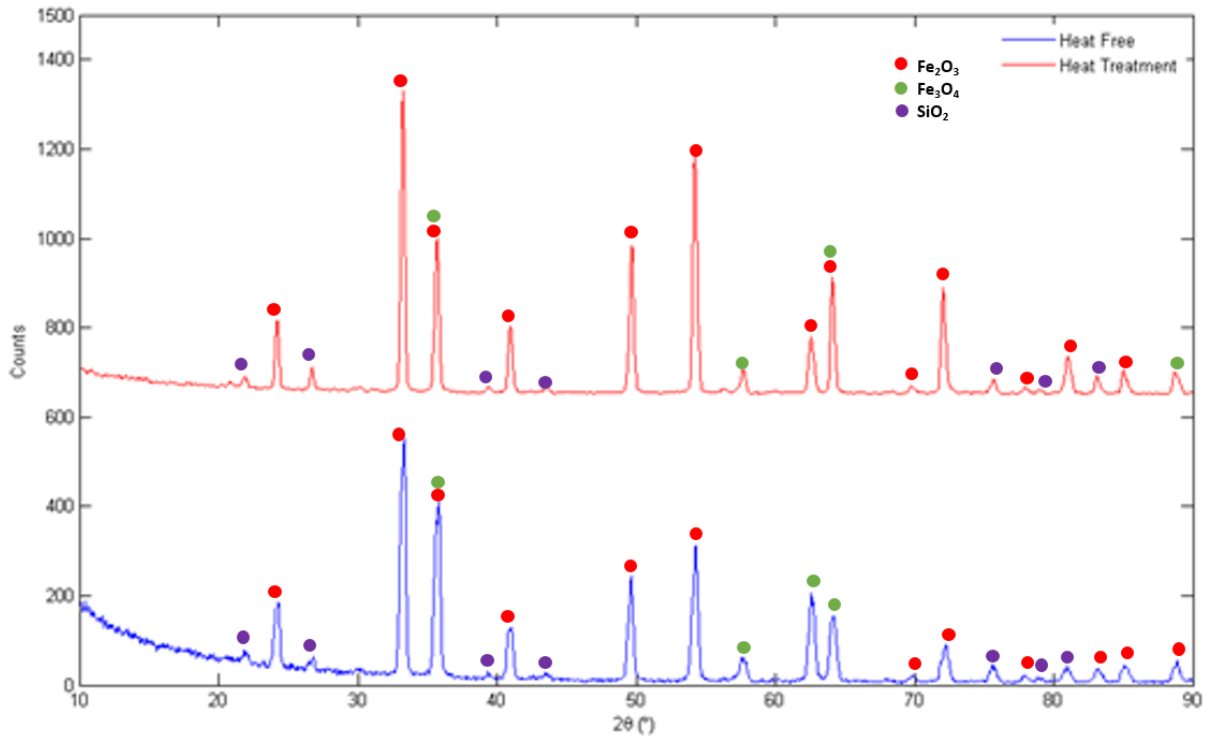
Mn	0.04	0.17	0.08
S	0.01	0.01	0.01
K₂O	0.06	0.01	0.08
Na₂O	0.09	0.10	0.10
P	0.01	0.01	0.01
Zn	0.01	0.02	0.01
Cu	0.04	0.04	0.04
As	0.01	0.01	0.01
Pb	0.01	0.01	0.01
TiO₂	0.05	2.78	0.06

The CaO/SiO₂ ratio is the ratio that helps us determine the basicity of the pellet. Looking at the XRF results, it was observed that all three pellets were acidic, with the east pellet at 0.34, the ferrexpo pellet at 0.11, and the domestic pellet at 0.28. TiO₂, which increases the viscosity of slag and pig iron at high temperatures, is present at a rate of 2.78% in the east pellet. The other two pellets contain TiO₂ below the desired level of 1%. The S, Mn, As, Cu, Zn, P, Na₂O, and K₂O content in all three pellets was found to be within the desired standards. The SiO₂ ratio, which causes slag formation, increases limestone usage when excessive, and reduces liquid iron yield (Sharma & Prakash, 1992; Shigaki et al., 1986; Sharma, 1994; Al-Kahtany & Rao, 1980), was observed to be lowest in the domestic pellet. Al₂O₃, which increases coke gas consumption when present at more than 1.5% in the pellet, was observed in the highest amount in the East pellet. MgO consumption increases when the Al₂O₃/SiO₂ ratio is greater than 1. The East pellet was found to be closest to this ratio. Therefore, it is thought that this is the reason why the MgO ratio is highest in the East pellet.

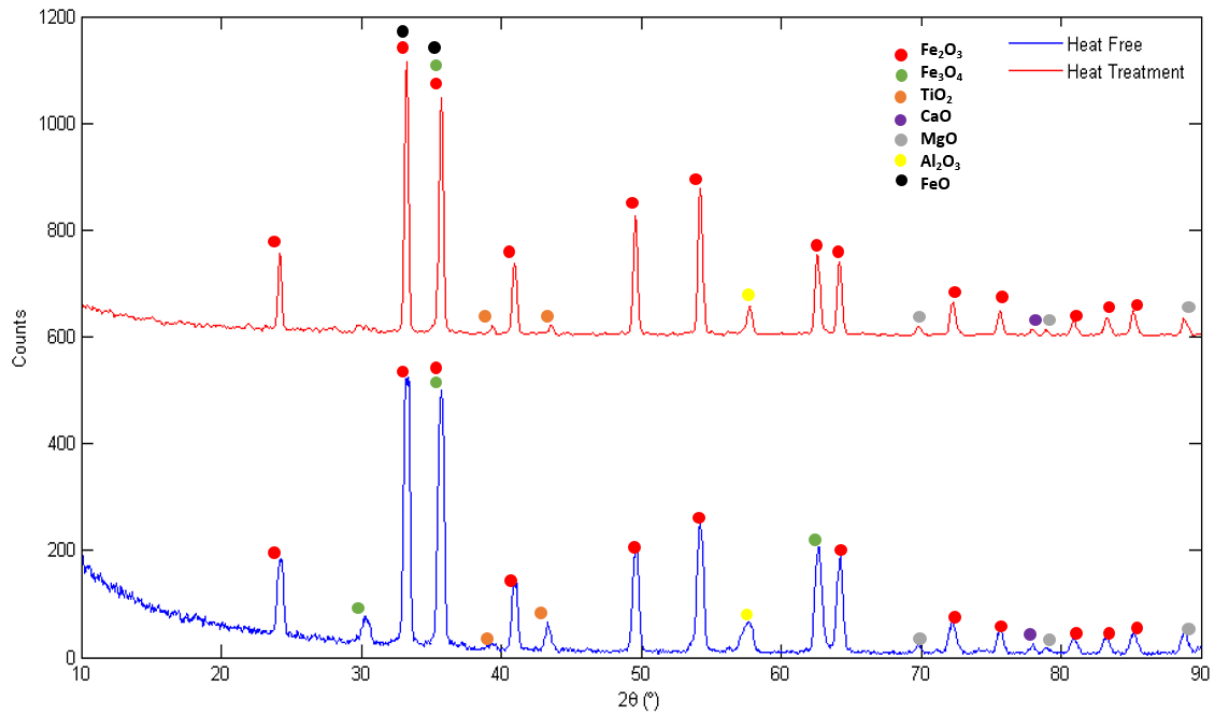
XRD patterns of heat-free and heat treatment for domestic, ferrexpo and east pellets are depicted in **Hata! Başvuru kaynağı bulunamadı.** (a, b, c), respectively.



(a)



(b)



(c)

Figure 3. XRD patterns of heat-free and heat treatment for **a)** Domestic, **b)** Ferrexpo and **c)** East pellets.

During the reduction of iron oxide, the final reaction occurs within the system through the mass transfer of the reducing agent and the removal of the product from the system, using either a gaseous or solid reducing agent. The chemical reaction rate is largely dependent on the solid contact area, which is determined by the porosity of the pellet. Furthermore, as various authors have noted, it is generally accepted that this is likely due to volume changes caused by phase transition and/or the growth of reduced iron whiskers during the reduction of iron oxide. Higher porosity appears to provide space for the growth of iron whiskers (Sharma & Prakash, 1992; Shigaki et al., 1986; Sharma, 1994; Al-Kahtany & Rao, 1980). According to XRD patterns, heat treatment led to phase transformation of hematite.

3.2. Physical Properties of Pellets

Sieve analysis results are shown in Figure 4. Reference size was chosen 6.3 mm because if sizes of pellets are smaller than 6.3 mm, pellets can be disintegrated in blast furnace. Thus, gas permeability of system decreases (Sharma & Prakash, 1992). Sieve analysis results show that domestic pellet has best ratio which means to amount of pellets bigger than 6.3 mm.

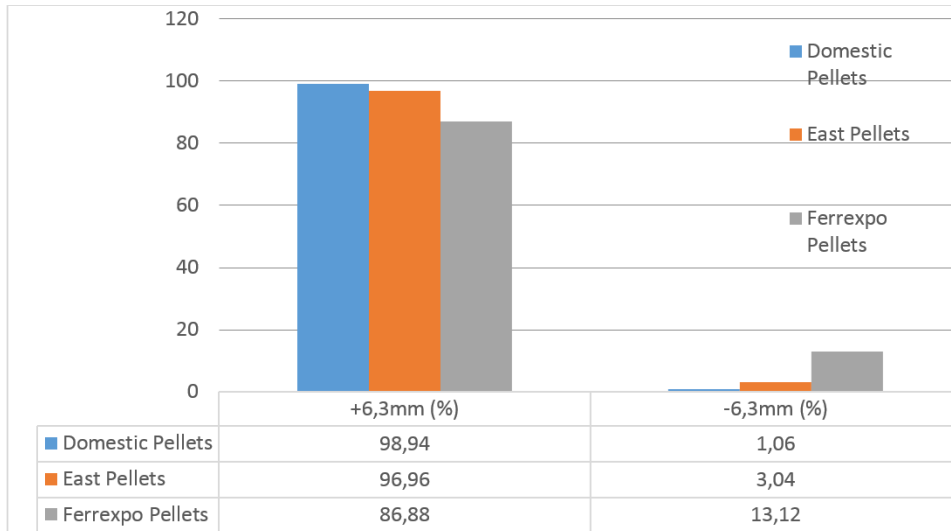


Figure 4. Sieve Analysis Results.

Usage of coke breeze increases with increase of moisture content of pellets due to moisture cause to decrease of peak temperature in blast furnace. Thus, lower moisture content is desired for pellets. Results show that domestic pellet has 0.24 wt.% moisture content. As shown in Figure 5, domestic pellet has lowest moisture content.

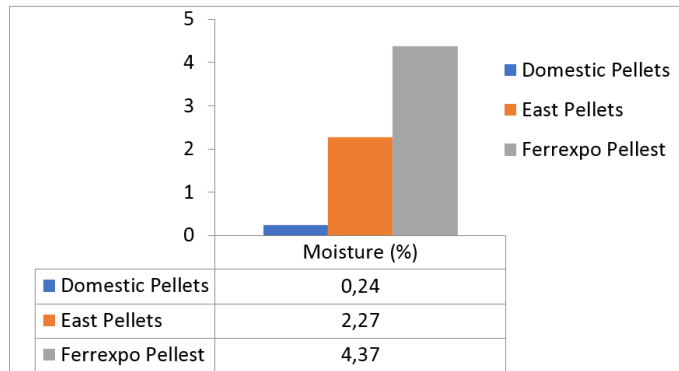


Figure 5. Moisture analysis results.

Abrasion index (A) of pellets should be maximum 5 for blast furnace technology in Kardemir A.Ş. According to this criteria, all pellet types are available for usage in blast furnace. However, ferrexpo pellet has lowest ratio compared to others. Another criteria is that disintegration index of pellets should be minimum 95 for blast furnace technology in Kardemir A.Ş. Disintegration index of east pellet is 93.92, so east pellet can not cover this criteria as shown in Figure 66.

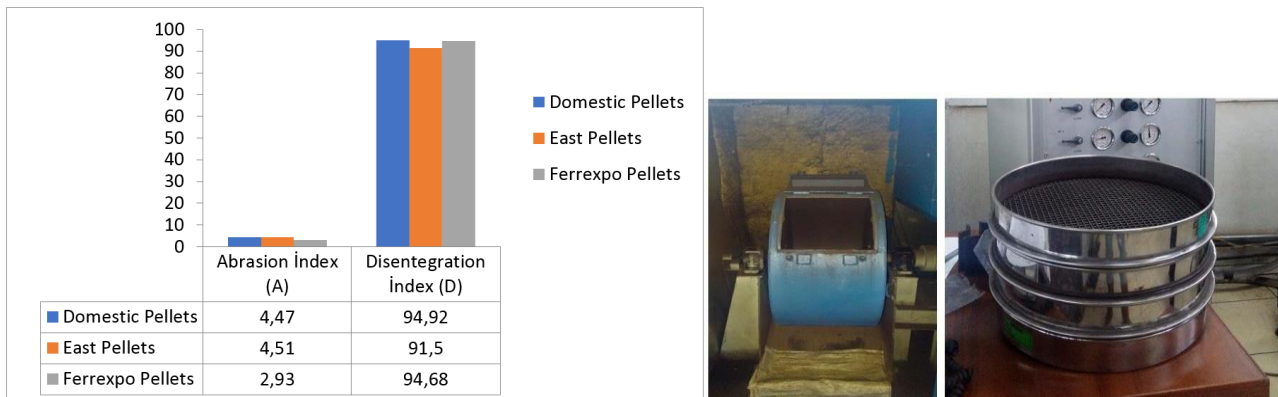


Figure 6. Tumbler test results, tumbler device and sizing screens after tumbler test.

3.3. Metallurgical Properties of Pellets

The RUL test provides information about improvements in pressure difference (dp) and height test (dh) results under load.

Dp: Pressure at the end of the reaction - pressure difference at the start of the reaction (1)

This formula helps us determine the dp value.

Dh : (reaction initial load - reaction end load) * 100/ the initial load of pellets in the tube (2)

This formula helps us determine the dh value.

RUL test results are shown in Figure 7. R40 is reduction speed at 40% reduction, dh is difference of height and dp is difference of pressure. Reduction speed is expected to higher than 0.75, and domestic pellet has best reduction speed. However, dp and dh values of domestic pellet are higher compared to imported pellets. Thus, domestic pellet is disadvantageous according to RUL test. Both imported pellets are available for usage in Kardemir A.Ş. blast furnaces. However, based on the results obtained, the metallurgical properties of the domestic pellet can be improved by taking into account the specific conditions listed below.

According to the results obtained here: Dp indicates the amount of pressure generated by high temperatures and gases in the blast furnace; Dp should not exceed 15 mm H₂O. Dh indicates the percentage of height lost by the ore in the blast furnace relative to its initial height.

Therefore, two important properties are sought in domestic pellets suitable for blast furnace charging: pressure resistance and porosity. The factors can be listed as follows:

- a) Size distribution and specific surface area: Increases in the percentage of fine particles and specific surface area increase pellet strength.
- b) Crystal shape in minerals: Pellets made from fine-grained and acicular-crystalline minerals are more robust than those made from other minerals (cubic, star-shaped).
- c) Moisture: Maximum strength wet pellets are obtained in mixtures containing only specific amounts of moisture in pellet rounding devices (drum, disc).
- d) Binders: Pellets baked with the addition of (Ca(OH)₂) and CaCO₃ increase pellet strength.
- e) Burning temperature and duration: Depending on the temperature and duration, some oxides and calcium ferrites form in the pellets, affecting pellet strength according to the formation status.

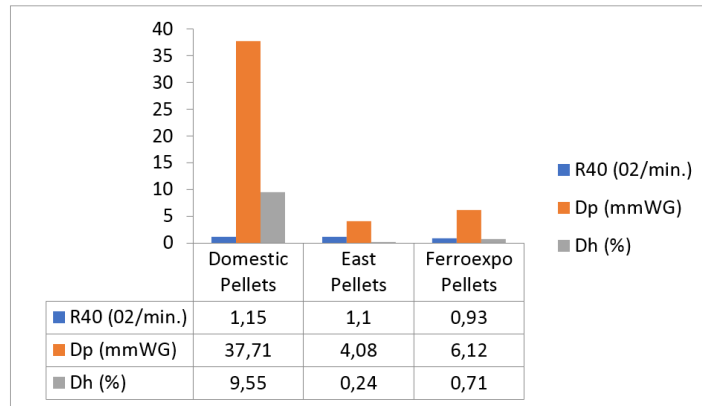


Figure 7. RUL Test Results.

According to Kardemir A.S. pellet specification, sizes of minimum 60 wt.% of pellets must be bigger than 6.3 mm while sizes of maximum 3 wt.% of pellets must be smaller than 6.3 mm. LTD test results show that all pellet types could not cover this criterion as shown in Figure 8. However, this metallurgical property for domestic pellet can also be improved. The (LTD) low-temperature reduction-crushing-dynamic test helps improve the determination of the ore's degree of crushing for the reduction of the pellet (20% CO + 20% CO₂ + 2% H₂ + 58% N₂) in a rotating tube at 500 °C (±5 °C). Accordingly, the reaction time for domestic pellets is 60 minutes; the reduction tube speed is 10 revolutions/min. The material exiting the tube is screened through 6.3-3.15-0.5 mm sieves. In the results obtained: +6.3 mm should be at least 84% and -0.5 mm should be at most 3% for metallurgical properties to improve. Furthermore, these results help determine the amount of coke consumed until the iron ores used as domestic pellet charge material in the blast furnace are converted into liquid pig iron; determine the collapses that will occur in blast furnaces due to pressure differences, height losses, and crumbling; and in determining which blast furnace iron ores to procure and prepare (purchasing and sinter preparation strategies).

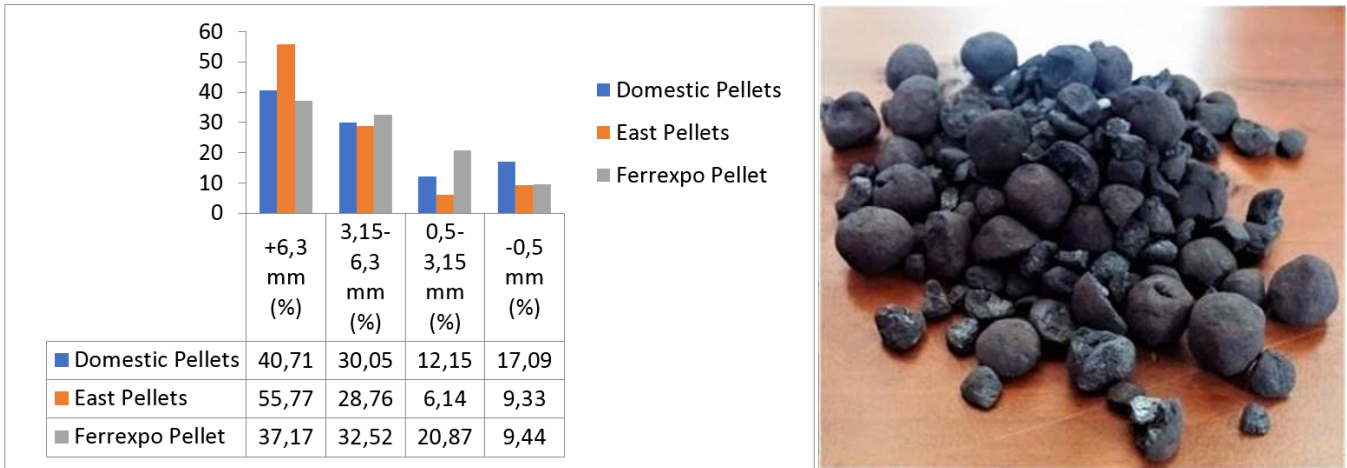


Figure 8. LTD Test Results, and LTD Test Application Post-Pellet Images.

The swelling index measurement, expressed as a percentage, of the volume increase occurring during reduction, based on the amount of gas absorbed by the pores, as determined by the swelling test applied to pellets, reveals the importance of porosity for the reduced form of iron oxide (Sharma & Prakash, 1992; Shigaki et al., 1986; Sharma, 1994; Al-Kahtany & Rao, 1980). According to Kardemir A.S. pellet specifications, the reference value for the free swelling index of pellets should be a maximum of 20%. Accordingly, when the FSI test results are examined, the fact that it is below 20% for all pellet types indicates as shown in Figure 9 that they exhibit optimal porosity.

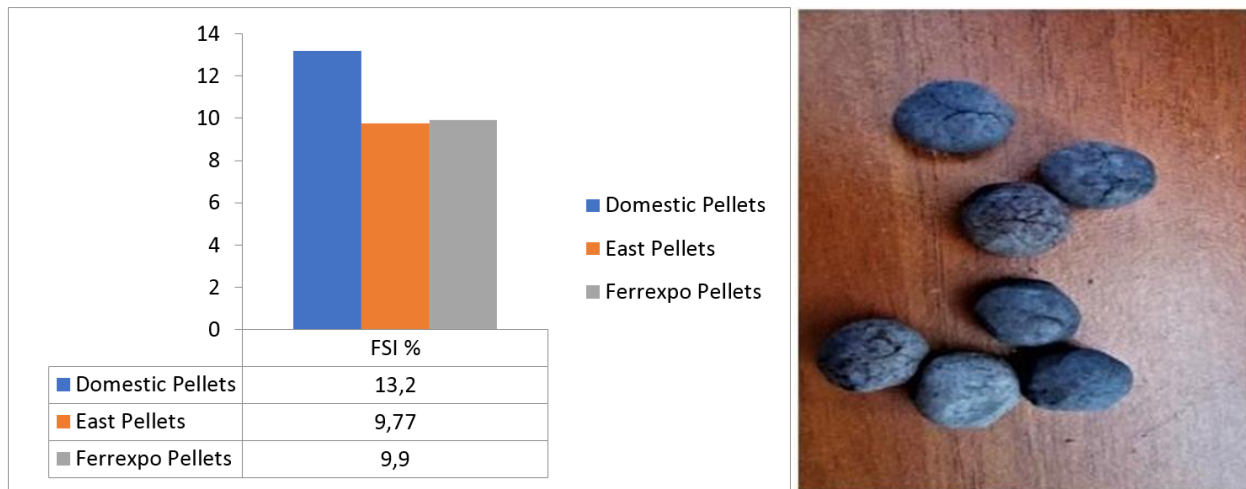


Figure 9. FSI test results and pellets subjected to FSI testing.

4. Results and Discussion

Surface morphologies, compositional and crystallographic structure analysis results for heat-free and heat treatment Ferrexpo, East and domestic pellets have been examined in detail. According to XRF and XRD analysis results, domestic pellets have the highest iron content (66.23%) and the best composition for blast furnace technology. The SiO₂ ratio, which causes slag formation, increases limestone usage and reduces liquid iron yield when excessive; this ratio has been observed to be lowest in domestic pellets at 2.24%. Furthermore, according to physical and metallurgical analyses, domestic pellets have the best reduction rate. If the pellet size is smaller than 6.3 mm (reference size), the pellets may break down in the blast furnace and the system's gas permeability will decrease. According to the sieve analysis results, the domestic pellet is larger than 6.3 mm and has the best ratio at 98.94%. The fact that domestic pellets have the lowest moisture content of 0.24% by weight reduces the use of coke dust in blast furnaces and provides economic benefits. Namely, sieve analysis concluded that domestic pellets have better gas permeability compared to imported pellets. The ore or pellets to be charged into the blast furnace must be wear-resistant, load-bearing, and not excessively dusty until the moment of reduction. This increases gas permeability and also improves iron quality. The wear index of all pellet types can meet the expected value. However, the wear index (A) of 2.93 for Ferrexpo pellets is well below the reference wear index value of 5 for the blast furnace technology at Kardemir A.Ş. The low-temperature fragmentation index of all pellets is lower than the expected value, and the pellet fragmentation index must be at least 95 for the blast furnace technology at Kardemir A.Ş. The fragmentation index of East pellets is 93.92, which does not meet this criterion. According to RUL test results, although the 40% reduction rate is higher than the reference value of 0.75

for all pellets, the higher dp and dh values of domestic pellets compared to imported pellets reveal the disadvantage of domestic pellets. That is, when the reduction time of carbon to oxygen is between 15 and 25 minutes, carbon-containing pellets meet both the metallization rate and strength requirements, and the metallization rate is above 80%. However, at high temperatures above 900 °C, significant volume swelling and low strength have been observed in carbon-containing pellets, which negatively affects multi-layer loading and heat transfer efficiency in blast furnaces.

Acknowledgments

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