PRODUCTION OF PELLETS FROM HEKİMHAN SIDERITIC IRON ORE FINES

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By
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SIDERITIC IRON ORE FINES

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ABSTRACT

In this study possibility of production of pellets from uncalcined Hekimhan sideritic iron ore fines, of size -20 mm has been investigated. The ore fine has been crushed and ground to desired size and pelletizing studies were made.

Influence of specific surface area and moisture content on pellet strength has been determined. Effects of indurating temperature, indurating time, and cooling rate on properties of pellets have been determined. Pellets were also produced using a 50 % Hekimhan sideritic iron ore fine and 50 % Divriği Dumluca iron ore fine mixture and properties of these pellets were also determined.

Key words : pelletizing sideritic fine , pellet production , pelletizing iron ore.

HEKİMHAN SİDERİT DEMİR CEVHERİ TOZUNDAN PELLET ÜRETİLMESİ

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ÖZET

Bu çalışmada -20 mm boyutundaki kalsine edilmemiş Hekimhan siderit cevheri tozundan pellet üretilebilme olanakları araştırılmıştır. Toz cevher kırma ve öğütme işlemleri ile istenilen boyuta indirilmiş ve pelletleme çalışmaları yapılmıştır.

Özgül yüzey alanının ve nem içeriğinin pellet sağlamlığı üzerine olan etkileri tespit edilmiştir. Pişirme sıcaklığı, pişirme süresi, ve soğutma hızının pellet özellikleri üzerine olan etkileri de belirlenmiştir. %50 Hekimhan siderit cevheri tozu ve %50 Divriği Dumluca cevheri tozu karışımından da pelletler üretilmiş ve bu pelletlerin özellikleri de belirlenmiştir.

Anahtar kelimeler : siderit tozu pelletleme , pellet üretimi , demir cevheri pelletleme.

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TABLE OF CONTENTS

Page

LIST OF TABLES

LIST OF FIGURES

1. INTRODUCTION

There exists a sideritic iron ore reserve of 88 million tons with a layer of oxidized ore lying above it in Hekimhan, Malatya. The oxidized ore has been used for the manganese needs of Turkish blast furnaces for many years and it is estimated that the quantity remaining is about 4.5 million tons. In addition to these, there exists a stock of 2 million tons of fine ore in the area(1).

As with any ore, there are basically two routes to follow in using this siderite ore in blast furnaces. The first alternative is charging the ore in lump form into blast furnaces. The second route is crushing and grinding -and perhaps concentrating- the ore and then charging it into blast furnaces after a suitable agglomeration process. The sideritic ore of Hekimhan contains about 36.5 % Fe and if it is to be charged into blast furnaces in lump form it would be appropriate to calcine the ore before charging so that transportation costs would be decreased and the adverse effects of calcination in the blast furnace would be eliminated. A study(2) has been conducted at MTA on the calcination of Hekimhan sideritic iron ore and there exists a feasibility study(3) on this subject. According to this feasibility report, the calcination plant will have an operational production capacity of 2 million tons. The ore will be crushed prior to calcination and the -20 mm portion will not be calcined. According to the project, the quantity of the -20 mm portion which will not be calcined is 500.000 tons/year. An agglomeration process will be necessary to use this iron ore fine as a source of iron.

In the evaluation of Hekimhan sideritic iron ore by using in blast furnaces, agglomeration therefore becomes important process in both of the routes mentioned above. Agglomeration is practically made by two methods. One of them is sintering, which is used for relatively coarser materials, such as feeds in size of 3-8 mm and the other is pelletizing that is commonly used for recently concentrated fine materials such as feeds in the size of -100 μm.

The possibility of using Hekimhan sideritic ore in blast furnace after sintering has been studied at MTA(2). It has been found that the sinter produced from only Hekimhan fine ore did not have sufficient strength due mainly evolution of CO_2 resulting from calcination during sintering. Sintering studies have also been made with a mixture of Hasan Çelebi fine and Hekimhan fine. It has been found that good quality sinter could be produced from this mixture.

In this study, the possibility of producing pellets from Hekimhan sideritic iron ore fineshas been investigated. Studies have been made on the fine stock of 2 million tons in Hekimhan.In the production of pellets the feed uncalcined fineshas been used, and calcination was aimed to take place during induration heating. The results indicate that good quality pellets can be produced from Hekimhan sideritic iron ore fines.

2. LITERATURE SURVEY

The production of pellets includes firstly the production of green pellets, and then the production of indurated pellets obtained by firing of green pellets. The production of green pellets is commonly made in practice by(4-6) , 1)disc pelletizers, 2) cylindrical drum pelletizers and 3)multiple cone drum pelletizers. The process of indurating of green pellets is generally made in(5-11), 1)shaft furnaces, 2)the straight grate furnaces and 3)the grate-kiln furnaces,

Indurated pellets should have certain physical properties so that they may be charged into blast furnaces. The most important two properties of pellets are compression strength and porosity. The parameters influencing these properties are as follows:

1)grain size distribution and specific surface area of the material,

2)crystal forms of the iron ore minerals and other minerals in the ore,

3)moisture,

4)binder addition,

5)indurating temperature and time,

6)cooling rate,

These parameters are examined below.

1.Grain size distribution and specific surface area of the material:

In general pelletizing is practiced for materials of particle size mostly under 100 microns,but max. particle size may be 0.5-1 mm. Specific surface area depending on grain size distribution has great effect on the compression strength of pellets. Strength of pellets increases with raising ratio of fine particles in material or specific surface area.

In the experiments of Merkhi and Devaney(12), coarse iron ore was balled. They found the values in Table 1 for the Wabush mix-a mixture of specularite and magnetite concentrates- (81.66 % -210 μm) and taconite concentrates (79.97 % -44 μm). The taconite concentrate and Wabush mix were balled by a mixture of binders of 0.7 % bentonite and 0.075 % soda ash ($NaCO_3$) and a certain amount of moisture.

Table 1. Physical Characteristics of Prefired Balls.

	Wabush mix.	Taconite concentrate
Greenball compression strength	2.60 lb	2.73 lb
Dryball compression strength	17.80 lb	28.24 lb
Average no of 18 in.drops withstood	5.3	7
Surface area (cm^2/gm)	749	1634

According to these authors, the strength of Wabush mix wet balls was lower than that of taconite concentrate balls. The reason for this was that the Wabush mix had a lower specific surface area than the taconite concentrate. The max. particle size does not have big importance, but the grain size distribution has a significant influence on the strength of wet balls.

The influence of the grain size distribution, especially the amount of finest fraction of the balling feed, on the strength of wet balls has been investigated by Urich and Han(13). They ground their specimens until 65, 70, 75, 80, 85, 90, 95 and 100 % of the specularitic iron

concentrate passed 325 M (44 μm), and prepared pellets with addition of 1.5 % bentonite and a certain amount of water. They found a relationship between the wet strength of the balls and the finest fraction percentage of balling feed as shown in Figure 1.

As seen from Figure 1, while the amount of -44 μm fraction increased, the wet strength of balls increased due to increasing specific surface area and hence raising number of contacts between the particles.

In the experiments of Burghardt et al(14), a number of iron ores were mixed and ground for a certain time and balled. The results showed a similar relationship mentioned above, as illustrated in Figure 2.

2. Crystal Forms of Iron Ore Minerals and other Minerals in the Ore

Crystal form of the ore mineral does not have an important effect on the wet strength of the green balls. Crystal forms of the ore mineral affect the specific surface area and the contact numbers between the particles which are bound by the effect of capillary pressure of the liquid.

Burchard et al(15) investigated the influence of the crystal forms of the iron ore minerals on the green strengths of the balls. They made synthetic iron oxides and Fe-hydroxides to observe the effect of the crystal forms in spherical, cubic or needle shape and in the form of star and fine distributed parts.

They had balled the synthetic iron oxides and - hydroxides by a certain amount of water with a defined liquid - filling degree. The wet strengths of these balls are shown in Figure 3 against the liquid-filling degree ψ in %.

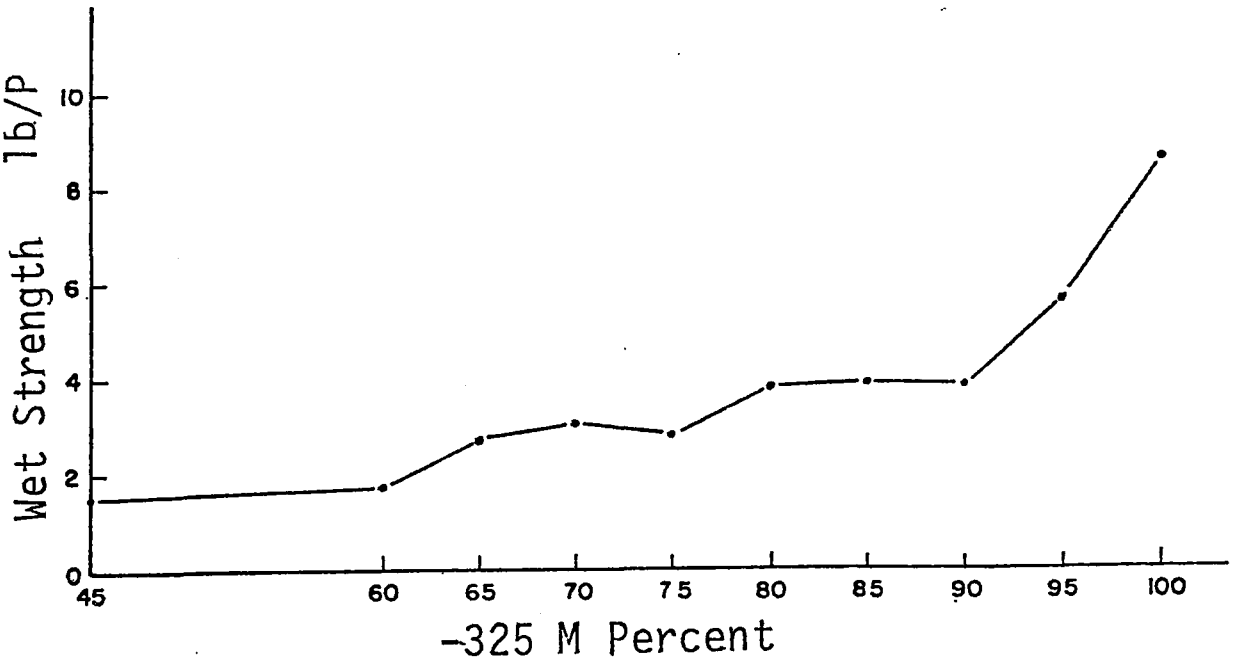


Figure 1. Percent -325 M in Concentrate vs Wet Strength of 1/2 in. Pellets.

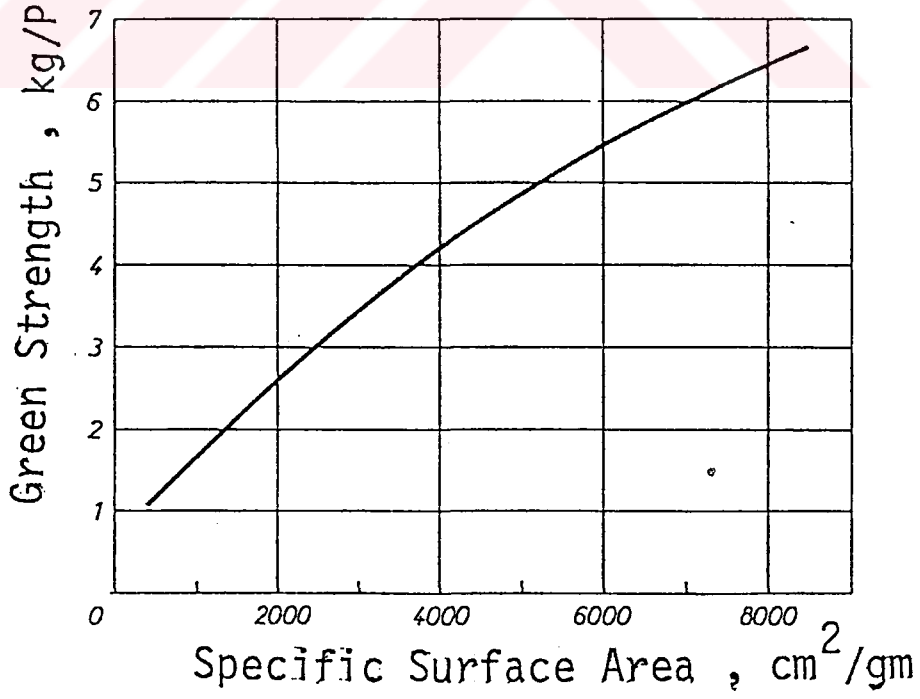


Figure 2. Dependence of Green Strength on Specific Surface Area.

As seen from Figure 3, at liquid filling degree less than 60% green strength of the balls with fine crystalline distribution of spherical crystals is greater than those of the pellets produced from needle crystals, cubic crystals, and star-form crystals. Further maximum green strength is obtained with fine crystalline distribution of spherical particles. Optimum liquid filling degree resulting in highest green strength is lowest with fine crystalline distribution of spherical particles and highest with needle-like crystals.

3.Moisture Content of Balls

Every balling process is made by a certain amount of liquid to obtain a good balling action. The liquid used is water or water containing a convenient quantity of binder. Moisture content influences the wet strength of the balls significantly. There is an optimum moisture content resulting in high green strength. This optimum moisture content of balls depends on specific surface area, surface conditions of the particles, micropore content of the particles of the balling feed.

The wet ball diameter is affected by revolution speed of balling machines and moisture content. As shown in Figure 4(16), the wet ball diameter increases with increasing moisture content and revolution speed of balling machine.

As seen from Figure 5(17) the wet strengths of balls significantly deperdonamount of moisture. The curves are seen to exhibit a maximum in a certain moisture range. The strength at a given moisture content as well as the optimum moisture content are seen from Figure 5 to depend on specific

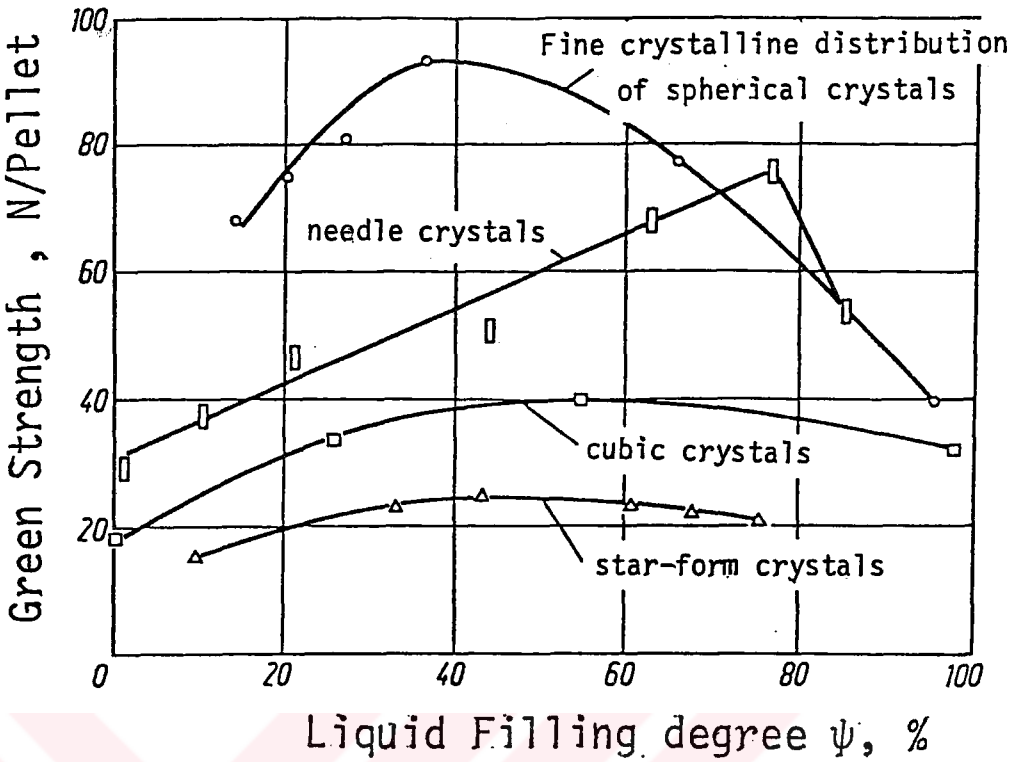


Figure 3. Green strengths of Pellets Produced from Synthetic Iron Oxides and -hydroxides vs liquid filling degree.

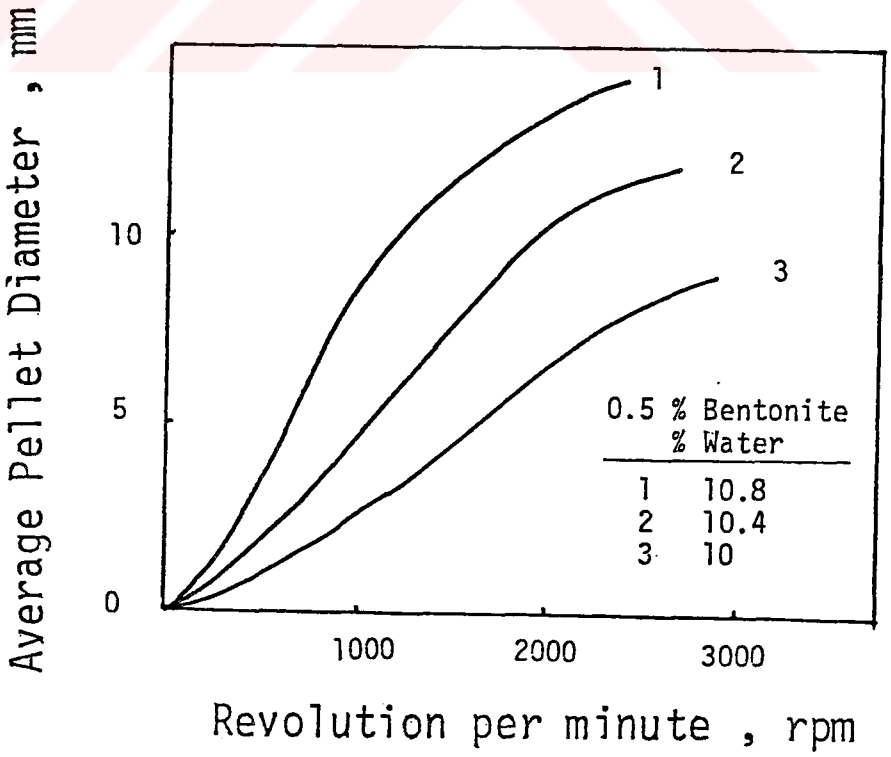
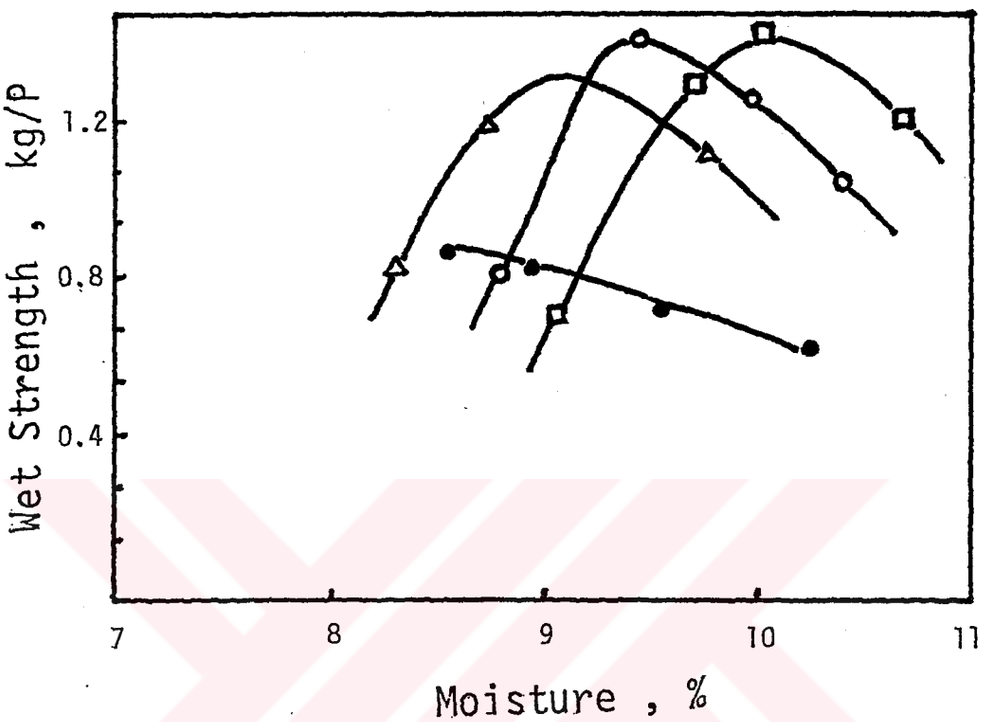


Figure 4. Influence of Water Addition on Growing of Green Pellets of Taconite Concentrate.



Specific Surface Area , cm^2/gm	-40 µm , %
● 1340	43.6
△ 1840	67.2
○ 2030	84.0
□ 3280	89.0

Figure 5. Influence of Moisture Content Over Green Pellet Strength.

surface area. As illustrated in Figure 5, optimum moisture content of 8.5 % of feed in size distribution of 65 % - 325 M was raised to a moisture of 11 % by yielding a feed of 95 % - 325 M. Optimum moisture content is seen to increase with increasing specific surface area.

4. Binder Addition

The binder addition is made for the following reasons:

(1)making pellet production easier,

(2)increasing green and dry strengths of balls,

(3)avoiding the problems during drying of the wet balls,

(4)obtaining products in good quality at low indurating temperatures,

(5)developing the properties of indurated pellets.

The binders may be divided into two groups: (1)organic binders and (2)inorganic binders

(1)Organic Binders:

Organic substances such as starch and dextrin have been tried as binders. It has been found that they affect the strength of pellets negatively. For this purpose they are not used as binders.

(2)Inorganic Binders:

Inorganic binders may be divided into three groups: (i)soluble salts in water, (ii)bentonite and (iii)basic binders.

(i)Soluble salts in water

A lot of soluble salts recrystallize with evaporation of moisture and create solid bonds. KCl, NaCl, NaOH, Na_2CO_3, K_2CO_3, $CaCl_2$, $MgCl_2$, $MgSO_4$, $FeSO_4$ may be given in this group.

These salts are generally not expensive, but Na and K are harmful to

furnace refractories, and also gases resulting from chloride salts are corrosive.

(ii)Bentonite

Bentonite, used commonly as a binder, is a kind of clay which contains montmorillonite minerals. Due to its very fine mineral particles in its structure and ion exchanging characteristics, bentonite has the properties of high plasticity, binding effect and colloidal characteristics.

Bentonite controls the effect of water in balling and provides high green strentghs for balls. Diameters of growing pellets decrease as the amount of bentonite increases. The reason for this is entrapment of a part of water between bonding layers of bentonite. Increasing amount of bentonite addition may raise the required moisture content. But higher moisture content may cause greater plasticity, so bentonite is added in amounts of 0.5-1% of the weight of the ore.

Bentonite provides sufficient green and dry pellet strength and helps to generate slag bonds giving rise to increased strength after induration.

(iii)Basic binders

Use of basic binders such as limestone, lime, MgO, dolomite, colemanite, etc has increased significantly in the recent past. Basic binders bind SiO_2 and thus the flux addition into blast furnaces is decreased or completely eliminated.

Lime may be used in the form of CaO or $Ca(OH)_2$. It was found by Ball(18) that the green strength of the pellets was not significantly affected by 1% $Ca(OH)_2$, but the dry strengths of the balls was increased by a factor of 2.5.

Davison et al(19) added 0.5-1% bentonite into Normanby mix (-1/8 inch) They found that bentonite additions lowered the green strength of the balls from 5.3 to 2.4 lb, but increased the dry strength from 8.8 lb to 32.2 lb. The use of 1% bentonite addition was found to be inconvenient since it resulted in green pellets with undesirably high plasticity, even though dried pellet strength was high (48 lb).

In the experiments of Davison et al(19), burnt lime (CaO) was added at 2½ %, 5% and 7½ % level to give basicities of 0.7, 1.1 and 1.5,respectively These additions had no effect on the green strengths of the pellets produced, but the dry strengths were improved with increasing lime content (13.9 lb, 15.4 lb and 23.8 lb respectively).

Finely ground basic slag was added at 5%, 10% and 15% level to give basicities of 0.73, 1.09 and 1.32 respectively and an improvement in dry strength was obtained with the 15% addition. The results showing the influence of binders on the Normanby mix (-1/4 inch) are given in Table 2(19).

Carter et al(20) investigated the effects of addition of chemically pure alumina (Al_2O_3), silica (SiO_2) and lime (CaO) (100 % -53 μm) on the hematite pellet properties. They(20) found the variation of abrasion index(%), cold compression strength (kN) with firing temperature and Blaine index (specific surface area, m^2/kg) for various lime and silica contents as shown in Figure 6 and 7. Strength of fired pellets have been found to be increased with increasing amount of limestone and dolomite by Kanetkar and Gupta(21) also. Their results are given in Figure 8.

Table 2. Properties of pellets made from -1/4 in Normanby mix.

Mix	*Green pellets* Pellets +½in dia. Moisture, %	Green strength, lb	Dried strength, lb	Firing temp., °C	Diameter, in	Compression strength, lb
−¼in (−½in+⅜in pellets)	8·5	4·0	17·2	1250	0·42	179
−¼in	8·0	4·0	13·2	1300	0·62	424
−¼in+½% Bentonite	10·0	5·5	32·6	1300	0·66	380
−¼in+2½% Burnt lime	10·0	6·6	18·3	1250	0·61	582
−¼in+2½% Burnt lime	8·0	5·3	30·1	1300	0·60	560
−¼in+5% Burnt lime	7·0	5·3	28·4	1250	0·62	448
−¼in+5% Burnt lime	8·6	5·1	28·4	1300	0·67	582
−¼in+7½% Burnt lime	8·8	7·0	40·9	1250	0·62	314
−¼in+7½% Burnt lime	9·3	6·4	40·9	1300	0·66	650
−¼in+2½% BF slag (−⅛in)	8·0	5·3	28·0	1250	0·56	604
−¼in+5% BF slag (−⅛in)	8·0	3·9	22·8	1250	0·57	716
−¼in+2% Basic slag (fine)	8·0	8·1	27·2	1250	0·57	560
−¼in+4% Basic slag (fine)	8·5	8·3	27·2	1250	0·59	1075
−¼in+2% Basic slag 2½% Burnt lime	7·0	10·8	15·1	1250	0·53	604
−¼in+2% Basic slag 5% Burnt lime	7·0	10·8	15·5	1250	0·58	851
−¼in+Slaked lime −⅛in	9·7	6·0	32·4	1250	0·59	940
−¼in+Slaked lime −⅛in	8·9	4·2	32·1	1250	0·59	1008
−¼in+Dolomite −⅛in	9·3	8·1	33·5	1250	0·58	1218
−¼in+Dolomite −⅛in	8·2	5·1	18·0	1250	0·57	1207
−¼in+Doloma −⅛in	9·0	11·3	3·5	1250	0·60	999
−¼in+Doloma −⅛in	10·0	2·9	1·3	1250	0·58	863
−¼in+Limestone flour	8·3	7·6	44·0	1250	0·62	2030
−¼in+Limestone flour	7·5	8·6	41·0	1250	0·62	1000
−¼in+½% Adcol	8·0	11·3	37·4	1250	0·60	851
−¼in+¼% Adcol	9·0	7·5	31·2	1250	0·60	918
−¼in+½% $FeSO_4$	9·0	7·6	14·1	1250	0·62	650

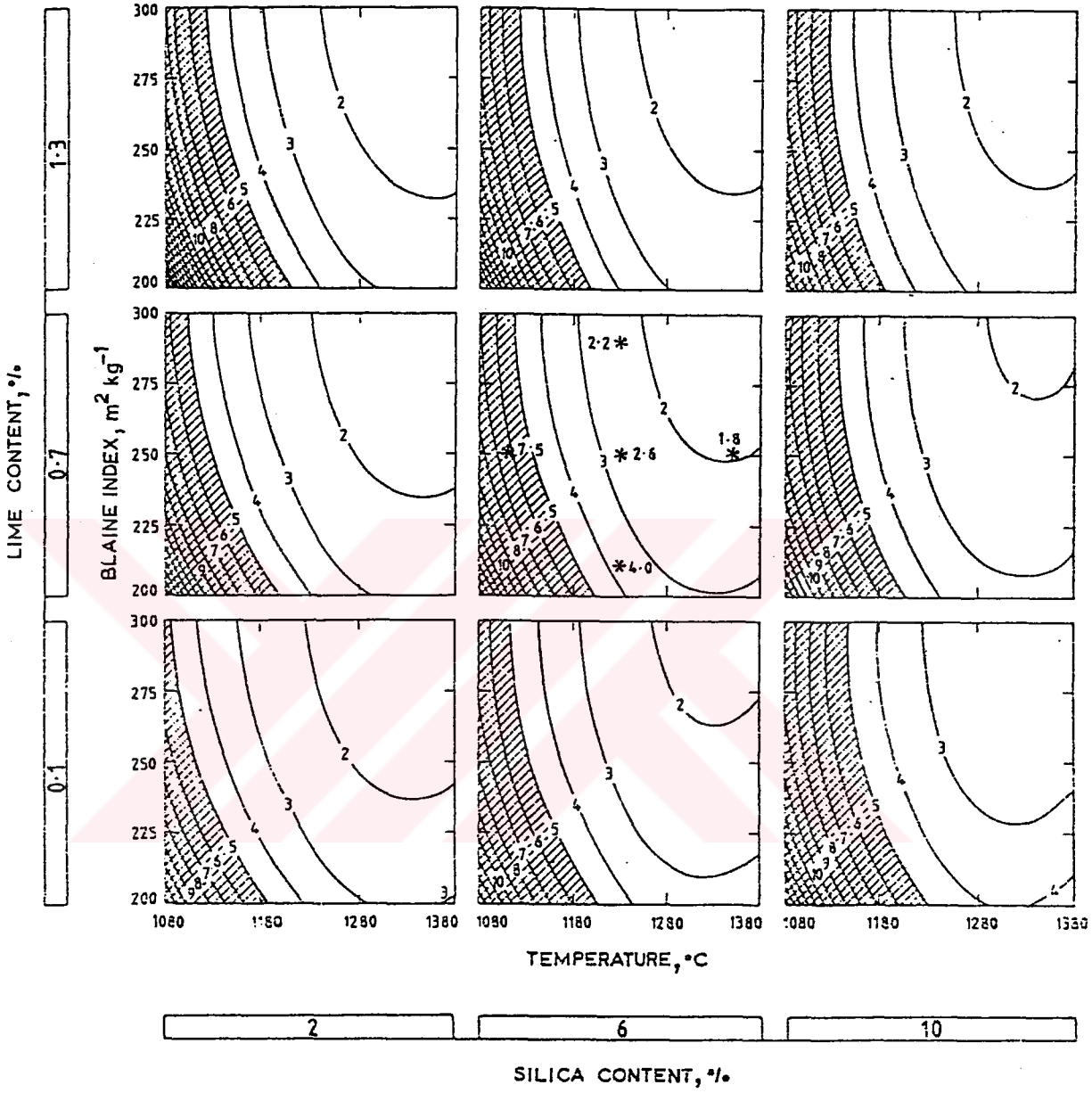


Figure 6. Variation of Abrasion Index, % , with Firing Temperature and Blaine Index for Various Lime and Silica Contents, Shaded Areas Show Values Failing to Meet Specifications.

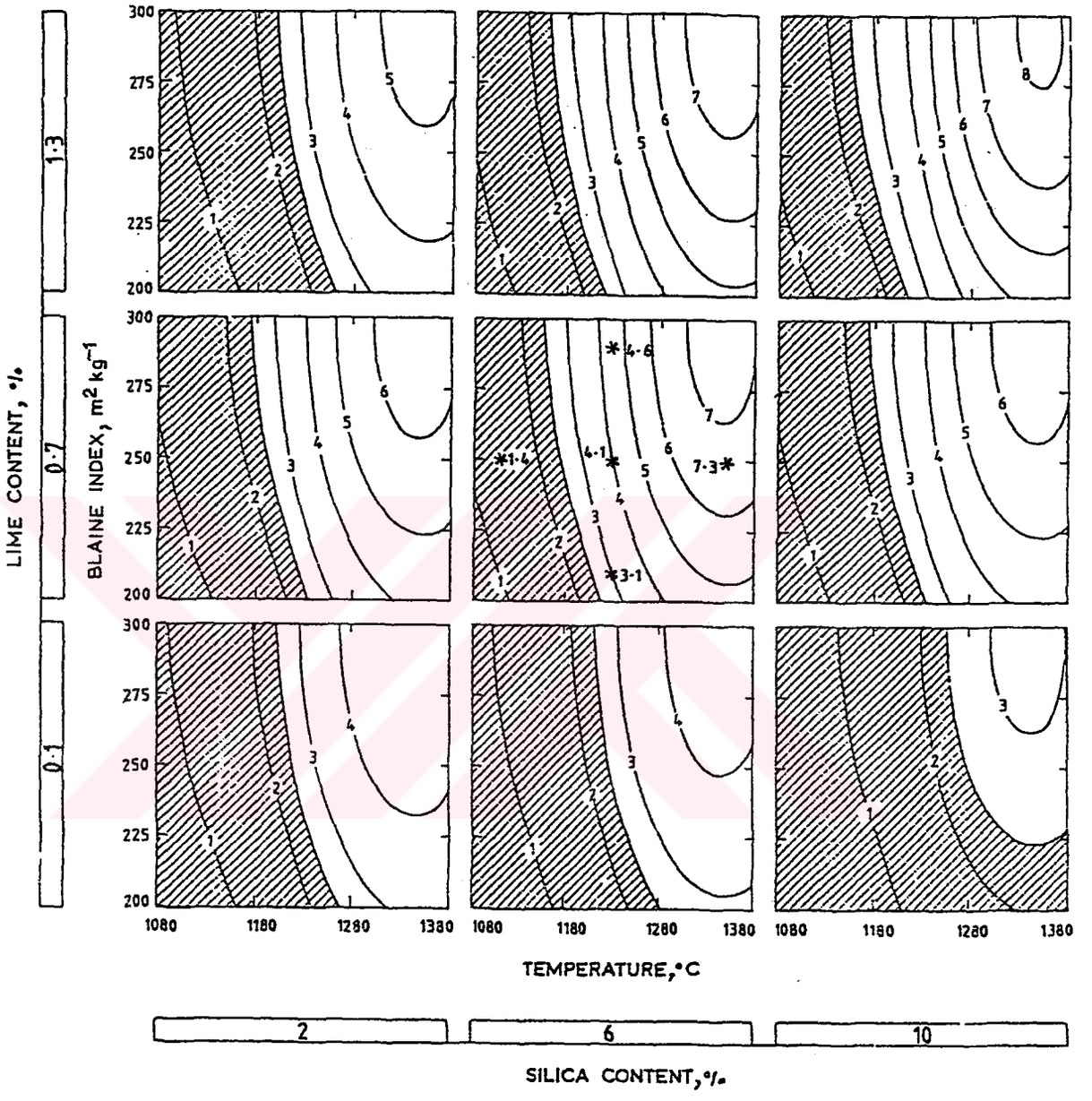


Figure 7. Variation of Cold Compression Strength, kN, with Firing Temperature and Blaine Index for Various Lime and Silica Contents, Shaded Areas Show Values Failing to Meet Specifications.

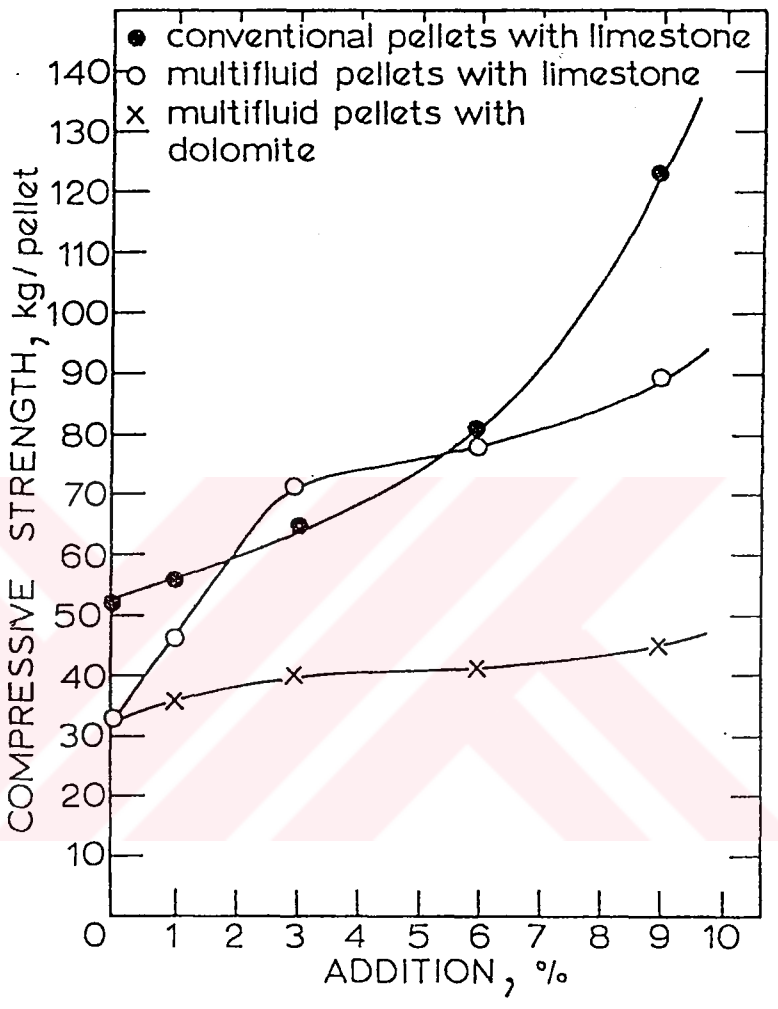


Figure 8. Variation in Compressive Strength of Fired Pellets with Addition.

According to Nekrasov et al(22) it was certainly understood that the components serving for melt formation are concentrate gangue (mainly quartz), the lime and magnesia of fluxing additives, bentonite and the ore minerals of magnetite and hematite. The non-metallic mineral components of the mix usually merge completely into the melt, and the ore mineral components to an extent of 4-7%.

They realized that basicity had a great influence on the temperature at which melt starts to form, on its viscosity, and on the mineral composition of silicate bond. Experiments showed that the lowest viscosity and the best glass formation capability is possessed by melts with a basicity of ~0.5. The most favourable for the strengthening process during firing was a basicity of the order of 0.5 (with a content of ~6-7% SiO_2). With a higher basicity the melt iron content fell , i.e. hematite and magnetite became less soluble in the melt.

Meyer(23), investigated the influence of $Ca(OH)_2$ and $CaCO_3$ additions and found that the compressive strengths of the pellets increased from 182 kg/p to 256 kg/p for 0.5 % $Ca(OH)_2$ addition, 354 kg/p for 3% $CaCO_3$ addition, and the abrasion index decreased from 8.7 % to 6.8 % for 0,5 % $Ca(OH)_2$ addition and to 2.7% for 3 % $CaCO_3$ addition (Figure 9 and 10).

Bojorski et al(24) studied the influence of basicity, on the crushing strengths and porosities of pellets of the siderite concentrate and found that the crushing strength of the pellets increased and the porosity decreased with increasing basicity.

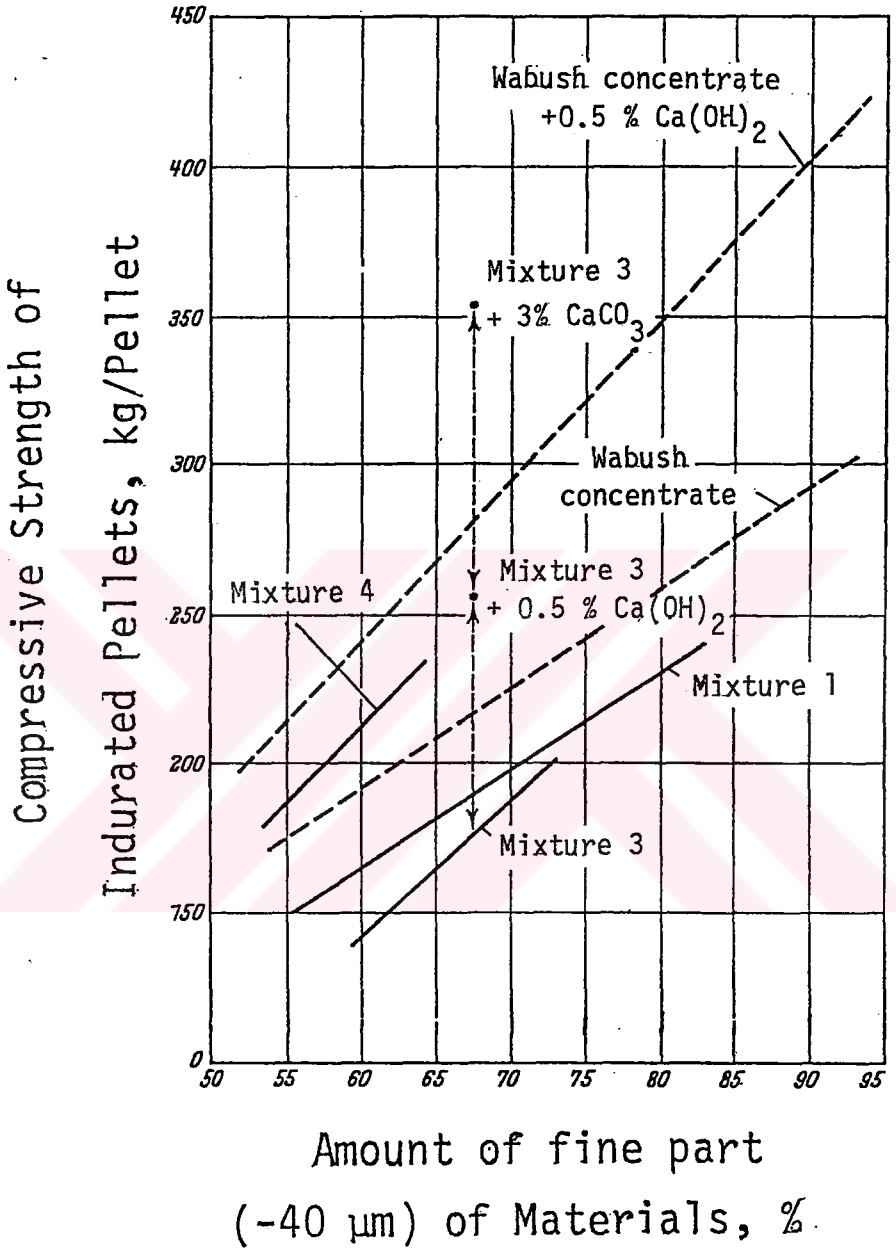


Figure 9. Influence of Binder Addition and Amount of Fine Part (-40 μm)of Mixture Materials on the Compression Strengths of the Pellets.

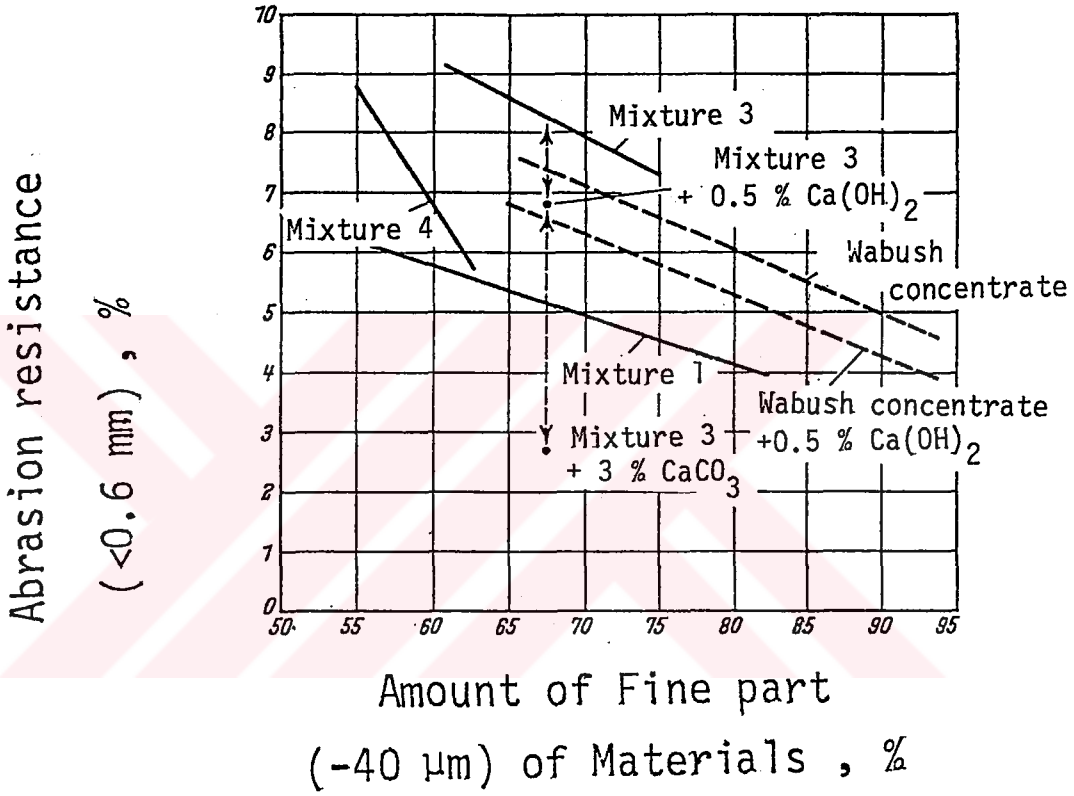


Figure 10. Influence of Binder Addition and Amount of Fine Part (-40 µm) of Mixture Materials on the Impact Resistance.

5. Firing Temperature and Time

Firing temperature and time has a significant influence over the physical characteristics of pellets. Optimum firing temperature and time are essentially dependent on the composition of the balls. Thus the constitutional study of iron ore agglomerates is of prime importance in determining the most satisfactory heat treatment to give optimum strength and reducibility.

Such features as the mineral content, type of bonding, microporosity, the mode of the formation of the minerals are significantly related with the blast furnace behaviour of the iron ore sinters and pellets.

Fitton and Goldring(25) tried to treat the constitutional changes resulting from independent variations in firing times and temperatures for distinct ore types. In their studies, a cleaner understanding could be obtained of the relation between heat treatment and pellet reducibility and strength.

The first kind, similar to the sinter mix used at Appleby Frodingham, consisted of calcareous Frodingham ironstone and siliceous Northants iron stone in the proportion of two to one, thus giving a self fluxing mixture as used on the sinter plant. The second kind similar to the sinter mix used at Workington, consisted of magnetite (Sydvaranger) concentrates and limestone with a small addition of burnt lime. A variation of this mix was prepared containing West Cumberland hematite fines. All the pellets were graded between 3/8 in and 1/4 in and were dried in air for 3h at $50^{o}C$ before firing.

The firing was done in air using a platinium-wound tube furnace, which was moved over the pellets to simulate the passage of the heat wave. Up to five pellets were fired at a time placed on a platinium wire support. The maximum temperatures used were between 1000°C and 1300°C and times ranged between 1 and 15 minutes. According to Fitton and Goldring the following results were obtained.

The mineralogy and microstructure of the fired pellets are similar to those normally present in better oxidized types of Workington and Appleby-Frodingham sinter. The principal iron-bearing minerals are magnetite, hematite and calcium ferrites and where silicate melt formation has occurred, these minerals are enclosed in a fine grained crystalline or glassy silicate matrix. In the absence of melt formation, the various original constituents, or calcination products, of the ores are present.

The extent of melting and the homogenization has caused some assimilation which would be significantly important for the final pellet characteristics as seen in Figure 11, it was realized that there could be an influence of the firing temperature and time on the constitutional parameters as the assimilation, softening, mineralogical structure of pellets.

(i)Softening

The single layer of pellets retained their original shape up to 1250°C, despite considerable internal melt formation. At about 1300°C there was some slagging and sticking of the pellets to the wire support.

(ii)Effect of hematite fines

At temperatures in the region of 1150°C , the hematite fines were

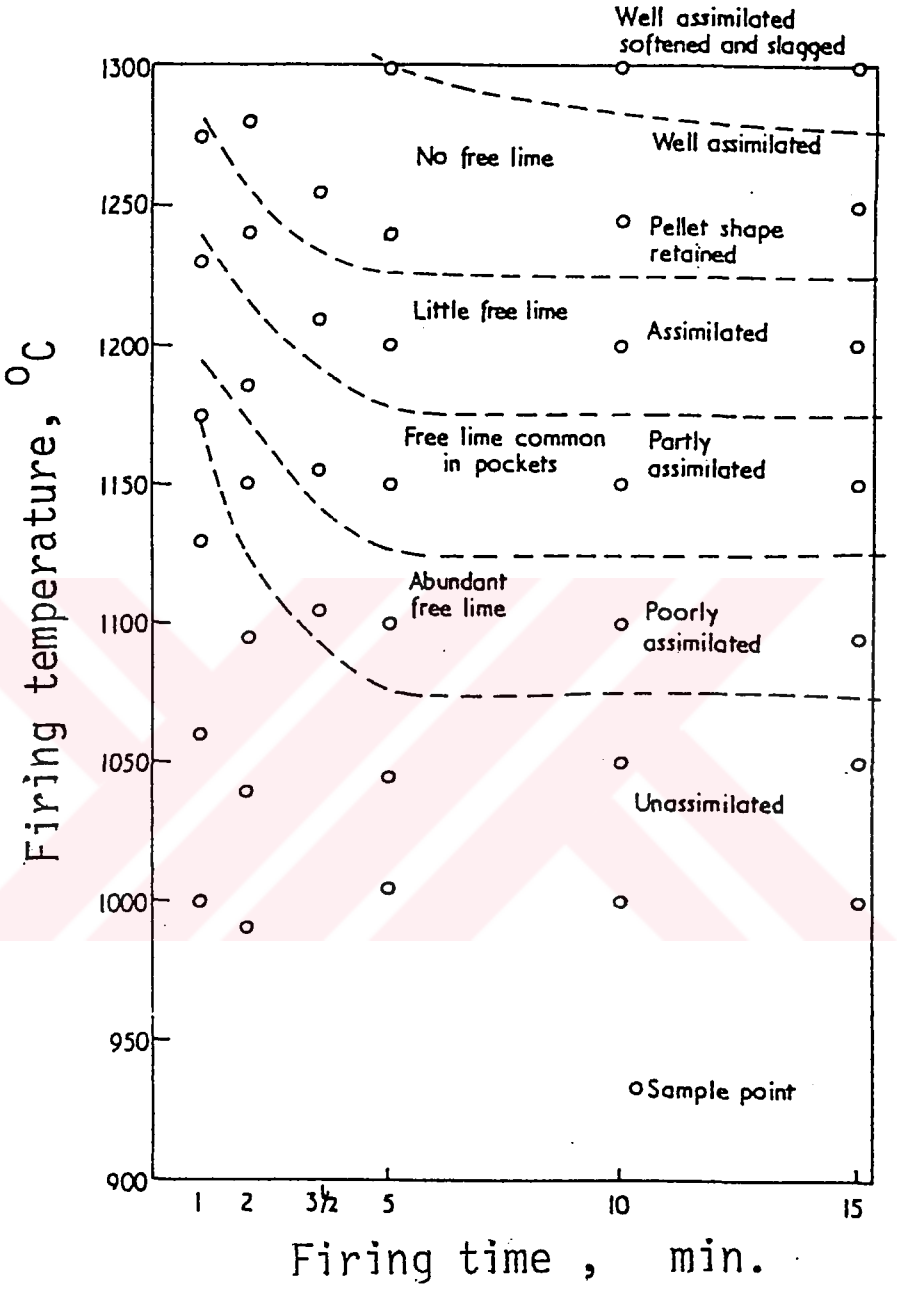


Figure 11. Variation in Extent of Assimilation for Appleby-Frodingham Pellets.

surrounded by virtually continuous pores, which separated them almost completely from surroundings. At temperatures towards and above 1200°C, the fines tended to become welded to their surroundings alongs parts of their peripheries. The development of this type of spherical pore could be expected to cause weakness in pellets of this kind.

(iii)Assimilation

Grades of assimilation of the slag-forming constituents might be characterized by definite changes in constitution (Figure 11) in particular with the occurrence of free lime in relation to time and temperature. These changes were defined as follows:

(i)Unassimilated ; No silicate melt has been formed between ore particles, although there may be some melt formation within individual calcined ore grains, particularly in the Appleby-Frodingham pellets.

(ii)Poorly assimilated ; small pockets of silicate melt have formed, entrapping the finer ore particles.

(iii)Partly assimilated ; There has been an unregular melt development. Finely crystalline calcium ferrites are common, due to a lack of homogenizatio

(iv)Assimilated ; The melt has developed and extended throughout the pellet, with the exception of some pockets relict from original constituents and comprising iron oxide, quartz, lime, etc.

(v)Well assimilated ; pocket of undigested particles are rare, although the mineralogy may not have become uniform. Pools of silicates, which appear at the assimilated stage, tend to be very well developed.

These groups tended to be temperature dependent (Figure 11) except at relatively short firing times.

(iv)State of oxidation

The oxidation of the pellets is topochemical in nature, the higher oxidized phases initially appear at the edges of the pellets.

The rate of oxidation appears to be related to the development of the silicate melt. At low temperatures (less than 1200^{o}C) when the melt develops more slowly, oxidation is relatively rapid. At higher temperatures (above 1250^{o}C) the silicate melt forms quickly and thereby impairs gaseous oxidation of the magnetite within it, and hence slows down the overall rate of oxidation.

(v)Formation of iron oxides and calcium ferrites

There are differences between some kinds of pellets with regard to the formation of iron oxides and calcium ferrites owing to the original ore constitution.

Fitton and Goldring(25) stated that there would be a strong tendency for the formation and recrystallization of magnetite, when the pellets of Appleby-Frodingham, contained magnetite and hematite originated from the calcination of the chamosite, siderite and limonite as iron ore minerals, were fired to a high temperature above 1250^{o}C with rapid silicate melt formation. For the same pellets, at temperatures in the range 1150 ^{o}C to 1250 ^{o}C, oxidation has taken place involving solution of magnetite into the melt and simultaneous crystallization of hematite and calciumferrites, particularly $CaO.Al_2O_3.2Fe_2O_3$, whose occurrence in alumina-rich sinters has also been demonstrated by Mazanek and Jasienska.

They realized that calcium ferrites appeared at a somewhat lower temperature for Workington pellets in which hematite was being formed by gaseous oxidation and in the temperature range 1100 ^{o}C to 1200 ^{o}C calcium

ferrites were common and precipitated from the melt.

Bojorski et al(24) made a study over the pelletization of siderite concentrates of the following percentage composition : 32.73 total Fe, 17.88 SiO_2, 3.20 Al_2O_3, 2.18 CaO, 1.53 MgO, 0.73 MnO, 0.28 P_2O_5, 0.12 TiO_2, 0.36 S, 24.58 ignition loss. They defined the constituents of the pellets with accordance to firing temperature as follows:

Firing temperature, ^oC	Phase Constitution		
1100 ^oC	Hematite	Fe_2O_3	
	Quartz	SiO_2	
	Ferrite	$CaO.2Fe_2O_3$	Small amounts
	Gehlenite	$2CaO.Al_2O_3.SiO_2$	
1150 ^oC	Hematite	Fe_2O_3	
	Ferrite	$CaO.2Fe_2O_3$	
	Quartz	SiO_2	
	Wallostonite	$-CaO.SiO_2$	Small amount
	Pseudo-wallostonite	$-CaO.SiO_2$	
	N-phase		
1200 ^oC	Hematite		
	N-phase		
	Pseudo-wallostonite		Small amount
	Wallostonite		
	Ferrite		
1250 ^oC	Hematite		
	N-phase		
	Pseudo-wallostonite		Small amounts
	Wallostonite		

Nekrasov et al(23) stated that the slag binder consisted mainly of an association of wallostonite with glass or with the ore mineral(hematite).

(vi)Bonding

The bonding of the mineral grains comprising pellets occurs in various ways according to the temperature of firing. These bonding variations are:

(i)Solid iron oxide bonding ; on oxidation of magnetite to hematite, a limited amount of bonding and bridging take place, particularly in the extremely fine-grained material (minus 0.005 mm diameter).

(ii)Recrystallization of iron oxides ; The continued grain growth of iron oxide crystals makes a large contribution to pellet strength, it has been considered that such grain growth takes place at temperatures above about 900^{o}C for magnetite and 1100 ^{o}C for hematite.

(iii)Bonding by calcium ferrites ; The formation of calcium ferrites by solid reaction of hematite and lime provides some bonding of the pellet structure.

(iv)Silicate bonding ; The silicate melt forms in the pellets on firing at 1100 ^{o}C - 1150 ^{o}C and, on cooling, chills to finely crystalline silicates or to glass.

Bojorski et al(24) realized that satisfactory properties were obtained from the pellets of siderite concentrate with silicate-phase bonding and binding properties of the phase might be affected by Al_2O_3 and MgO which were present in siderite concentrate.

The authors (25) believe that the aim should be to sinter or heat-harden pellets under as oxidizing an atmosphere as possible and for a certain time to yield a somewhat melt formation and iron oxide recrystallizati giving enough compressive strength.

Fitton and Goldring (25) determined that 5 minutes of firing time at 1150 ^{o}C to 1200 ^{o}C gave satisfactory results. (Optimum strength and reducibility).

6.Cooling Rate

Cooling rate has a big effect on the compressive strength of the pellets. Extremely high cooling rates lead to brittle glass formation and result in decreased strength.

It is stated by Nekrasov et al (22) that, depending on rate of cooling, the melt turns into a silicate binder with different phase compositions and structures, which in its turn sharply influences the metallurgical properties of pellets. And it is said by Butkarev et al (26) that during cooling from firing temperature of 700-900 ^{o}C the pellets pass from plastic into the elastic state. At the end of the cooling process, as temperatures equalize over the pellet cross-section, residual stresses arise in transition region, the levels of which are determined by size of the temperature difference ΔT between centre and surface of the pellet in the transition zone.

In the experiments made by Butkarev et al (26) it was found that the presence of cracks due to residual stresses reduced the cold strength of iron ore pellets. These authors investigated the effect of cooling rate on the cold strength of 10 mm diameter pellets fired at 1300^{o}C. The cooling rates in different experiments were 50, 100, 150, 200, 300 and 500 deg/min. Their results showing the effect of cooling rate over residual

stresses and cold strengths of hematite and magnetite pellets are given in Figure 12. Dependence of cooling rate limits of hematite and magnetite pellets on their radii are shown in Figure 13. As seen from Figures 12 and 13, cold strength of the pellets decreases with increasing cooling rate and cooling rate limits decrease with increasing pellet radius.

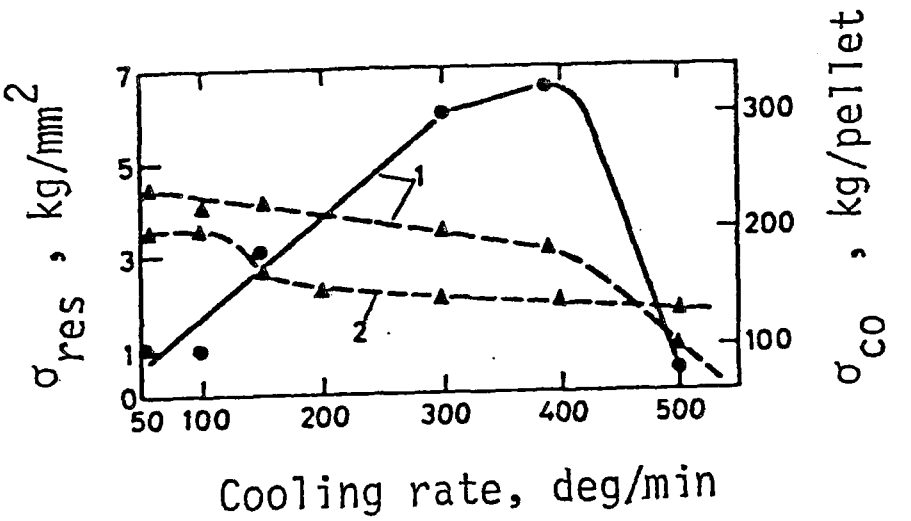


Figure 12. Dependence on Cooling Rate of (1) Hematite and (2) Magnetite Pellets of (——) Residual Stresses,(----) Cold Strength.

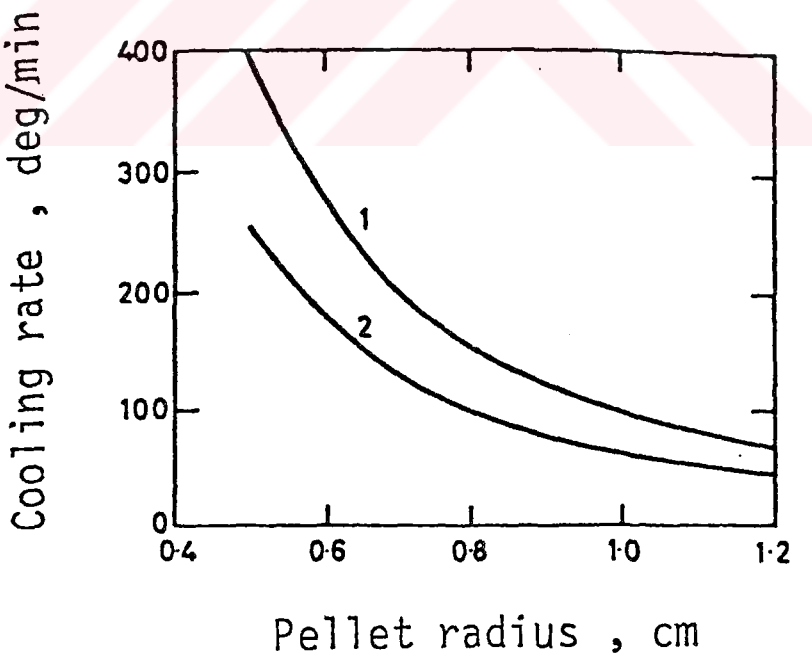


Figure 13. Dependence of Cooling Rate Limits of (1) Hematite and (2) Magnetite Pellets on Their Radius.

3. EXPERIMENTAL

3.1. Introduction

A 25 kg representative sample has been taken from Hekimhan sideritic iron ore finesand pelletizing studies have been made after the preparation process described below. The preparation process before pelletizing contains comminution of the fines to -0.5 mm with a few crushing and grinding stages and determination of grain size distribution of the finesand the ground sideritic finesand chemical analysis of the fines. The procedure followed in preparation of the finesis shown in Figure 14.

Cylindrical rather than spherical pellets were prepared so that dimensions, qualities and structures of the pellets produced would not be different. The cylindrical pellets were prepared by mixing of 3 gm fine with a certain amount of water, putting this mixture into a steel mold and then pressing. Pellets were pressed by a pressing equipment which has a maximum loading capacity of 2 tons. Effects of operating parameters on pellet properties were determined from studies conducted on cylindrical pellets and in accord with the results obtained from these experiments spherical pellets have been produced in a disc pelletizer. The diameter of the disc pelletizer is 1 m and it rotates along an axis inclined 45^{o} to the horizontal at a speed of 24 rpm.

3.2. Measurement of Pellet Properties

Compression strengths of the pellets were measured by compressing

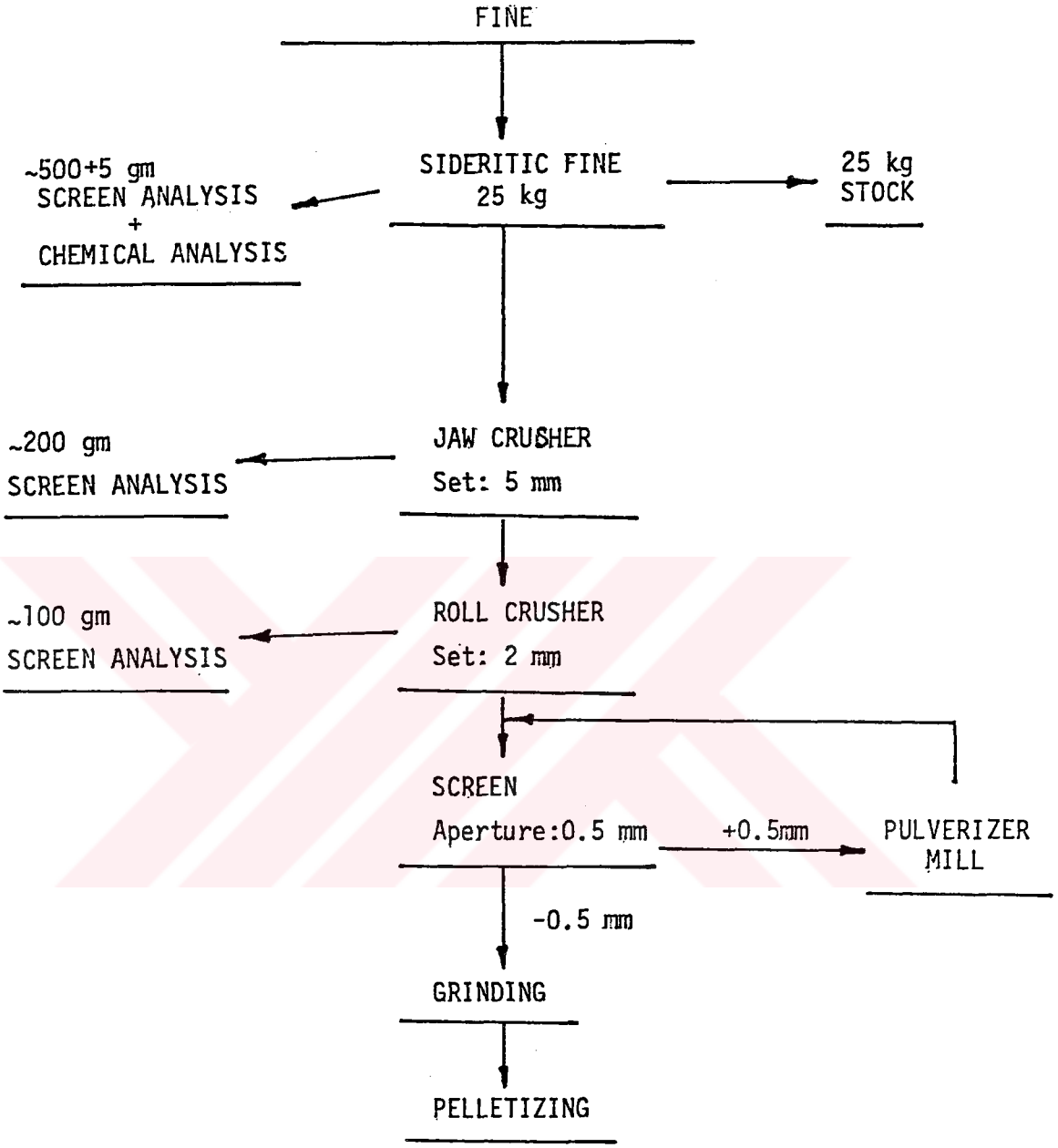


Figure 14. Flowsheet of Preparation Processes Before Pelletizing.

the pellets in a pressing equipment until the pellets are broken. The pressing equipment used has a maximum loading capacity of 2 tons and has a load gaugewith which the load applied can be measured,

Porosities of the pellets were calculated using the equation:

$$\varepsilon\% = (1 - \frac{\rho_{APP.}}{\rho_{T.}}) \cdot 100$$

where $\varepsilon\%$ is the porosity of the pellet, $\rho_{APP.}$ is the apparent specific gravity of the pellet and $\rho_{T.}$ is the true specific gravity of the pellet. Apparent specific gravities of cylindrical pellets were determined by measuring the dimension of the indurated pellets with a caliper and then calculating the volumes. Apparent specific gravities of spherical pellets were determined by using a mercury vacuum pycnometer. The true specific gravity of the pellets has been found by grinding the pellets to a particle size of -100 μm, and measuring the volume of the fines in a water pycnometer.

3.3. Determination of the Optimum Pelletizing Conditions

In this section, effects of moisture content and particle size distribution of the pelletizing mix, pressing load, induration temperature and time, cooling rate of the indurated pellets were studied and optimum pelletizing conditions were determined. Cylindrical rather than spherical pellets were used in the experiments for dimensional uniformity purposes. Experiments carried out are presented below under separate headings.

3.3.1. Determining Optimum Moisture Content for Green and Dry Pellets

A 100 gm- representative sample was taken from the specimen ground in disc mill. This sample was dried at 105°C and divided into 3 gm-parts for determining optimum moisture content. These 3 gm-parts were separately mixed with 6, 8, 10, 12, 14 % by weight of water, placed into a special cylindrical steel mold and pressed under a load of 150 kg. Compression strengths of the pellets were then determined in the green state and also after drying at 105°C for 2 hours.

3.3.2. Determining Optimum Pressing Load with Respect to Porosity and Strength

A 100 gm-representative sample was taken from a specimen ground in disc mill and then in a laboratory ball mill for 20 min. This sample was dried at 105°C and divided into 3 gm parts for determining optimum pressing load.

These 3 gm parts were mixed with 10 % water, placed into a cylindrical steel mold and pressed under loads of 50, 100, 150, 200 and 250 kg. The green pellets obtained were then subjected to the indurating process shown in Figure 15. A total of six pellets were prepared with each pressing load. Three of these pellets were crushed in a compression test equipment having a max. loading capacity of 2 tons. The other 3 indurated pellets were used to determine porosities in a vacuum pycnometer.

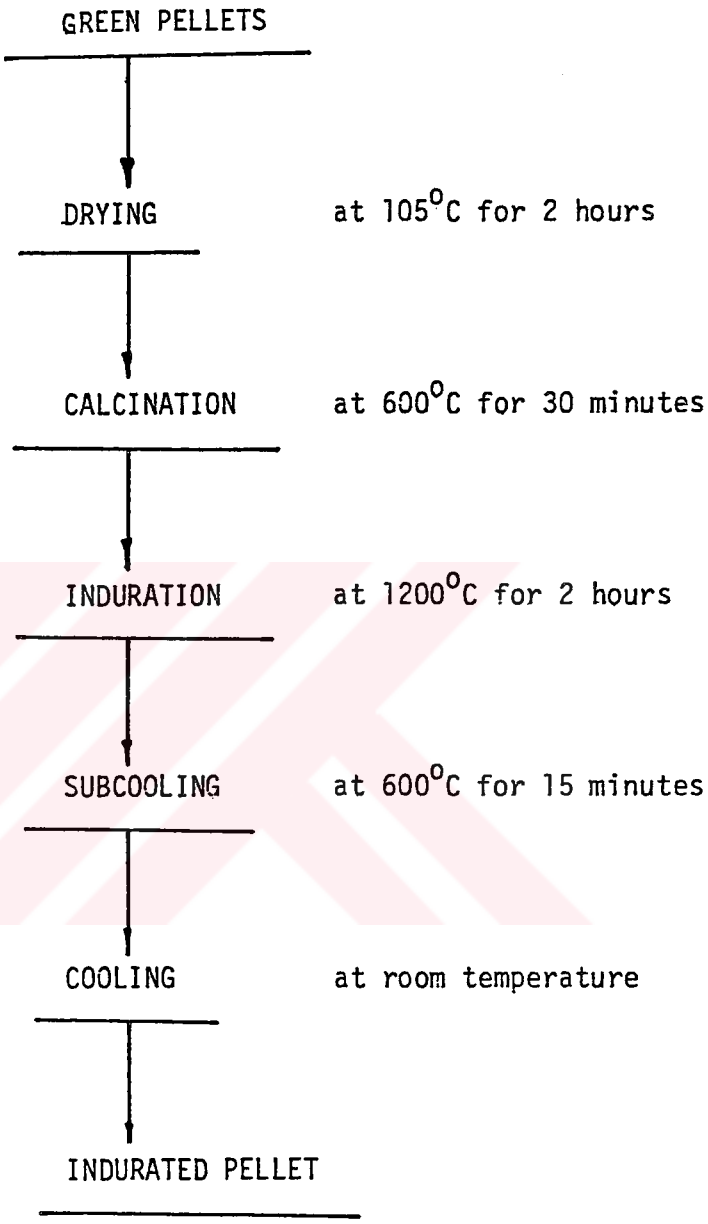


Figure 15. Flowsheet of Indurating Process of Green Pellets.

3.3.3. Determination of Optimum Particle Size Distribution and Optimum Induration Temperature,

The influence of particle size distribution of the ore-fine particles on pellet quality was investigated by using a 15 kg representative sample, This sample was divided into 5 equal parts and each part was subjected to grinding for different periods of time. One of the equal parts has not been ground and other parts have been ground for 20, 40, 60 and 80 minutes. Approximately 200 gm representative sample was taken from each group which was subjected to screen analysis. The results obtained from those tests are given in Table 3.

From each of these parts, 10 representative 3 gm- specimens were mixed with addition of 10% water, placed into a cylindrical steel mold and pressed under a load of 150 kg. Green pellets were indurated at $1000^{o}C$, $1100^{o}C$, $1150^{o}C$, $1200^{o}C$ and $1250^{o}C$, respectively. The other induration steps are similar to those in Figure 15. The crushing strengths and porosities of indurated pellets were measured and the optimum grinding time and firing temperature were found in taking into account that excessive grinding results in high energy consumption, and high induration increases the extent of fayalite formation.

The weights and porosities of the two pellets dried at $105^{o}C$ for 2 hours and calcined at $600^{o}C$ for 30mins. , and also indurated at above temperatures were measured. Thus the variation in weight loss and porosity of pellets produced from Hekimhan fine were determined.

Table 3. Screen Analysis and Specific Surface Areas of Hekimhan Sideritic Iron Ore Fine Ground for Certain Times.

Fraction (mm)	Not ground (-0.5mm)		Ground For 20 mins.		Ground For 40 mins.		Ground For 60 mins.		Ground For 80 mins.	
	Weight,%	Cumulative Undersize, %	Weight,%	Cumulative Undersize, %	Weight,%	Cumulative Undersize, %	Weight,%	Cumulative Undersize, %	Weight,%	Cumulative Undersize, %
-0.5+0.21	38.16	100	8.35	100	3.1	100				
-0.21+0.149	9.49	61.84	14.77	91.65	11.77	96.9				
-0.149+0.105	15.79	52.25	16.8	76.88	12.39	85.13	20.09	100	9.8	100
-0.105+0.063	11.19	36.56	19.75	60.08	10.44	72.74	12.77	79.91	12.7	90.20
-0.063+0.044	10.17	25.37	15.23	40.33	8.85	62.3	8.12	67.14	11.21	77.50
-0.044	15.2	15.2	25.1	25.1	53.45	53.45	59.02	59.02	66.29	66.29
specific surface area, $cm^2/gm.$*	600		900		1500		1620		1770	

*Specific surface area : $\frac{6}{\gamma_s}\left(\sum_{i=1}^{n}\frac{1}{(F_{i+1}+F_i)/2}\cdot\frac{w_i(\%)}{100}\right)$

γ_s: specific gravity of specimen (gm/cm^2)

F_i: size of fraction i (cm)

w_i: weight percentage of fraction i

3.3.4. Determining Optimum Induration Time

A 100 gm representative sample was taken from the part ground for 40 min. From this sample, 12 representative 3 gm specimen were mixed with addition of 10% water, placed into a cylindrical steel mold and pressed under a load of 150 kg. Green pellets were subjected to the indurating process shown in Figure 15, but pellets were indurated for different induration times and cooled in furnace. Induration time was changed from 2 minutes to 4 hours. The crushing strengths of the indurated pellets were ascertained and thus influence of induration time on strength was determined.

3.3.5. Determining the Effect of Cooling Rate on Strength of Indurated Pellets

A 100 gm sample was taken from the specimen not ground (-0.5 mm). In order to observe this effect, this representative sample was dried at 105°C for 2h. and divided into 3 gm-parts. These 3 gm parts were mixed with 10 % water, pressed under a load of 150 kg. These green pellets were then subjected to identical heating, but different cooling steps. The cooling processes employed were quenching the pellets into water, subjecting the pellets directly to air, placing the pellets into a pot furnace kept at 600°C, keeping them there for 15 minutes and then subjecting them to air, placing the pellets into a pot furnace kept at 600°C, keeping them there for 15 minutes, placing then into an oven kept at 105°C, keeping them at this temperature for 10 minutes and then subjecting to air.

3.4. Properties of Pellets Produced from Hekimhan Siderite Fine and Dumluca Magnetite Fine Mixture

Hekimhan sideritic iron ore fine and Dumluca magnetite fine were mixed in a 1:1 weight ratio, and pelletizing studies described above for siderite fines were conducted on this mixture.

3.5. Chemical and Microstructural Investigation of Hekimhan Sideritic Fine and the Pellets Produced from this Fine.

A 5 gm sample was taken from fine ground to -100 μm and this sample was subjected to chemical analysis. Chemical composition of Hekimhan sideritic fine is given in Table 4.

Thin section of fine was taken from a representative ore part in size of about 10 mm. A face of this specimen was polished by SiC powder and sticked with Canada balsame over a lamella and other face was then polished by SiC powder up to a thickness of 5 μm and mineral distribution was observed under a transmission microscopy.

A representative thin section of Hekimhan sideritic fine in thickness of 5 μm was observed by microscopical examination and the microphotograph of the thin section of this fine was taken.

Microphotographs of pellets were taken from a few representative pellets. These pellets were put into a resin and a face of this resin was polished by firstly Al_2O_3 powder then Cr_2O_3 powder and etched by HCl in a few second and observed under a reflection microscopy.

Table 4. Chemical Analysis of Fines

Components	Grade, %	
	Hekimhan Fine	Dumluca magnetite fine
Fe	32.2	52.9
Fe_3O_4	0.8	37.3
S	0.09	0.17
SiO_2	14.40	12.74
Al_2O_3	4.95	1.59
CaO	2.60	1.37
MgO	4.31	1.70
Mn	3.26	1.85
K_2O	1.03	0.36
Na_2O	0.28	0.18
Weight loss at 1200°C	16.9	-

4. RESULTS AND DISCUSSION

4.1. Influence of Moisture Content

The effect of moisture content on the wet and dry strengths of the pellets is shown in Figure 16. As seen from this figure the wet strengths of the pellets are in the 1-2 kg/cm^2 range. The optimum moisture content is seen to be 10% from Figure 16.

4.2. Influence of Pressing Load

The effect of pressing load on pellet properties is shown in Figure 17. The compression strengths of the indurated pellets are seen from this figure to increase linearly with pressing load. Pellet porosities are seen to decrease with pressing load.

Although the strength of the pellets produced under the highest pressing load is the highest, a pressing load of 150 kg was chosen to be used in preparation of the cylindrical pellets. This choice was based on the consideration that higher pressing loads would make the process approach briquetting. The strengths of the indurated pellets pressed under a load of 150 kg are sufficiently high (280 kg/pellet) and their porosities are about 39% which is much higher than that required for pellets to be charged into blast furnaces.

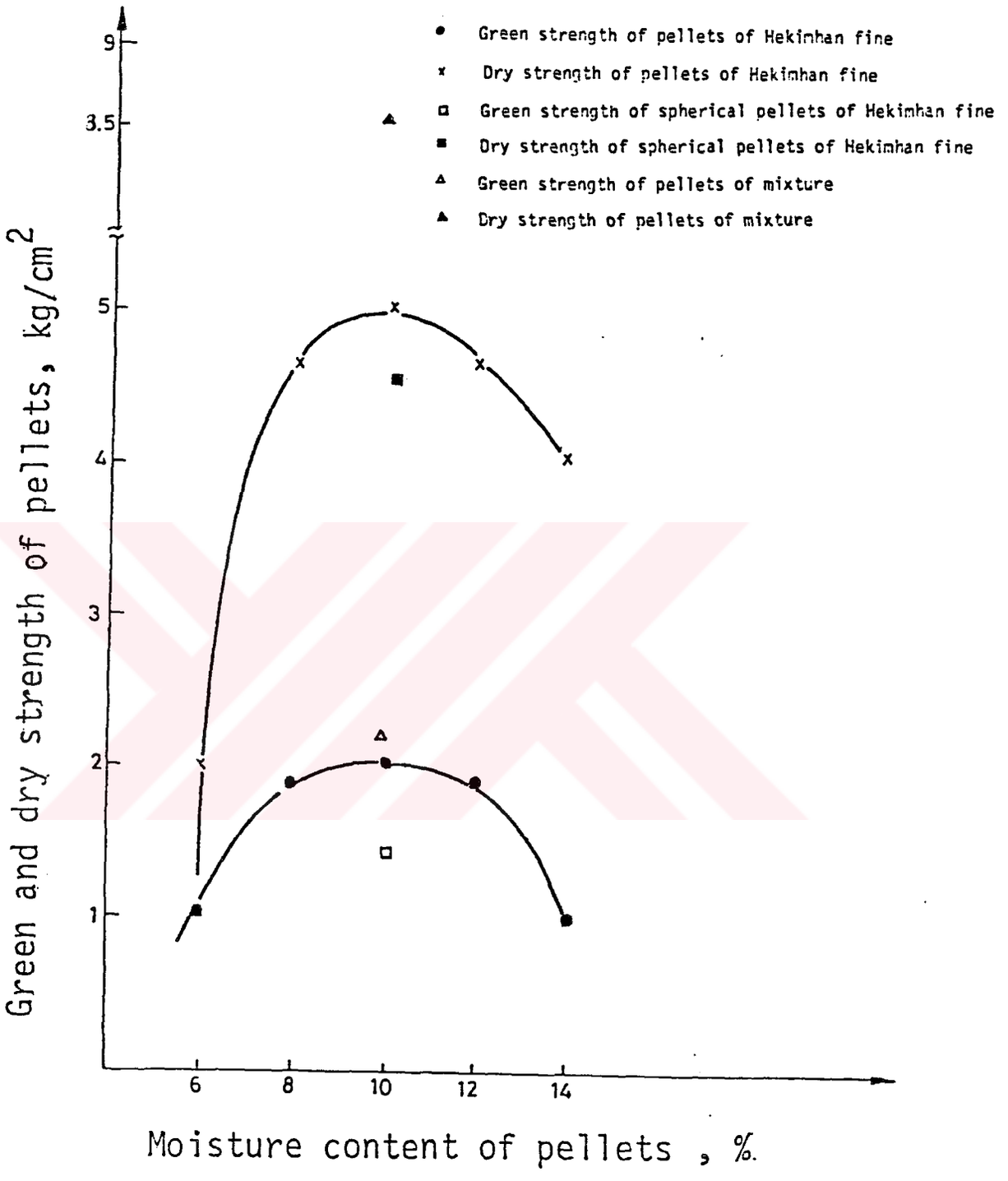


Figure 16. Variation in Green and Dry Strength of Pellets vs Moisture Content.

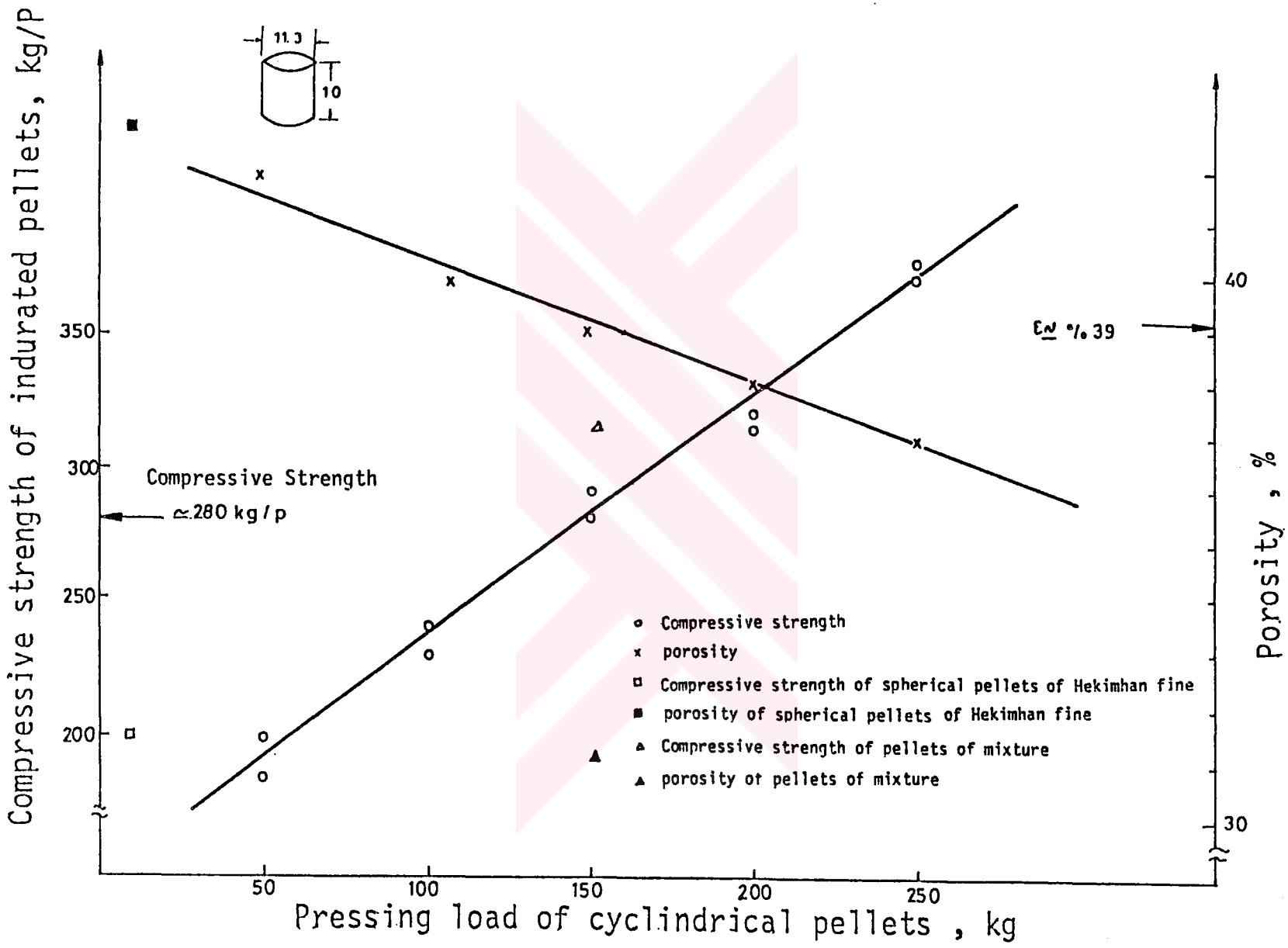


Figure 17. Variation in Indurated Pellet Strength and Porosity vs Pressing Load.

4.3. Influence of Particle Size Distribution and Indurating Temperature

The effects of particle size distribution or specific surface area of pelletizing fine and induration temperature on the compression strengths of indurated pellets are shown in Figure 18 a-b. It is seen from Figure 18 a-b that increasing grinding time or increasing surface area of the pelletizing mix increases the strength of the pellets. Optimum grinding time was chosen as 40 minutes because the strengths of the pellets produced from the fine ground for 40 minutes are sufficiently high and longer grinding times will give rise to increased costs.

The strength of the pellets are seen from Figure 18 a-b to increase by a factor of 2.5 as the induration temperature increases from 1100^{o}C to 1250^{o}C. 1200^{o}C was chosen as the optimum induration temperature. This choice was based on the fact that it was possible to attain a sufficiently high strength (200 kg/pellet) at 1200^{o}C. Increasing costs and increased likelihood of fayalite formation with increasing induration temperatures were also taken into consideration in choosing the optimum induration temperature.

4.4. Influence of Induration Time and Cooling Rate

The effect of indurating time on the strengths of pellets fired at 1200^{o}C is shown in Figure 19. As seen from this figure, the strength of the pellets increases very steeply with time within the first 15 minutes. A strength of 280 kg/pellet is reached at the end of 30 minutes which does

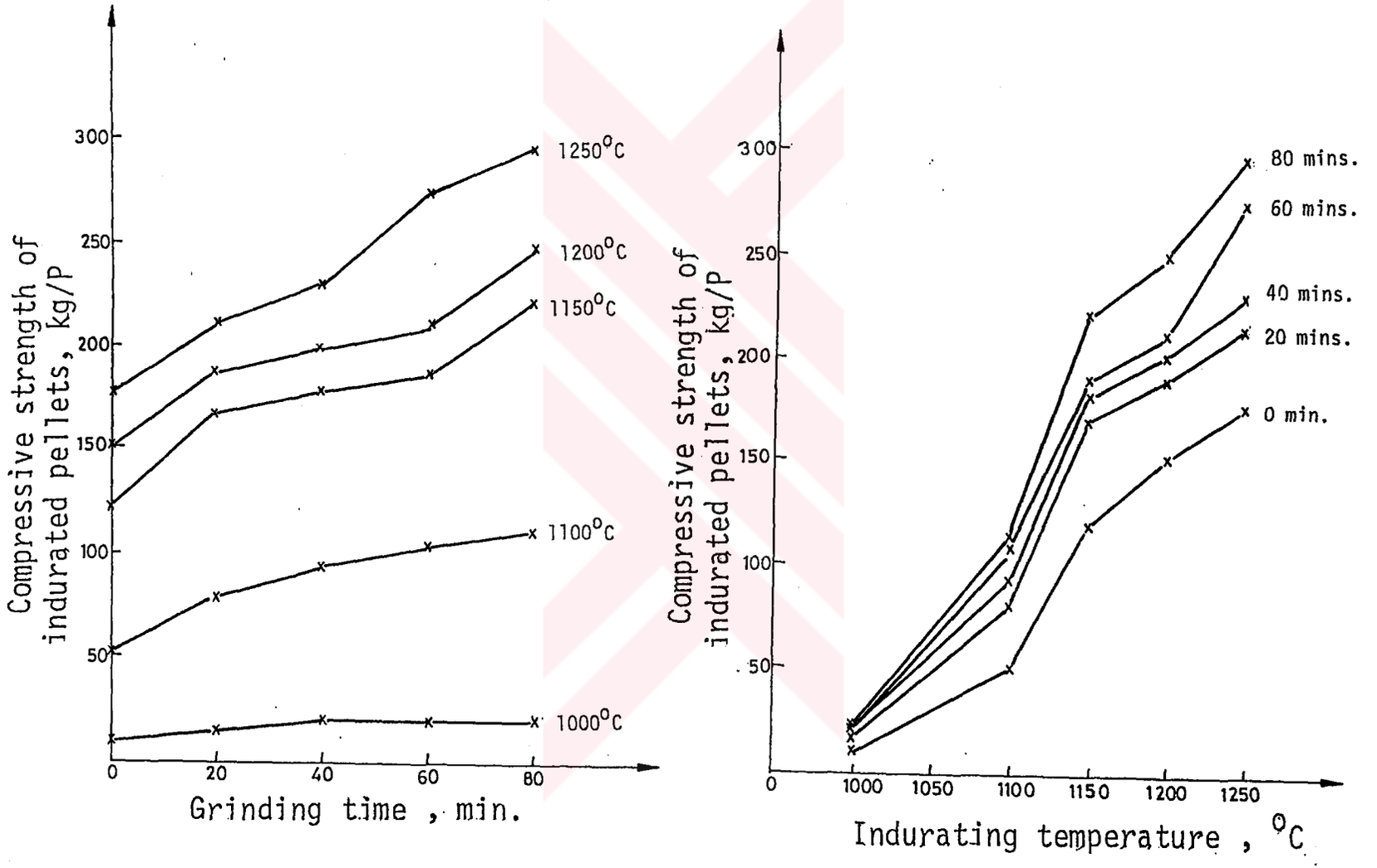


Figure 18a-b. Effect of Grinding Time (grain size distribution or specific surface area) and Indurating Temperature on Compression Strength of Indurated Pellets.

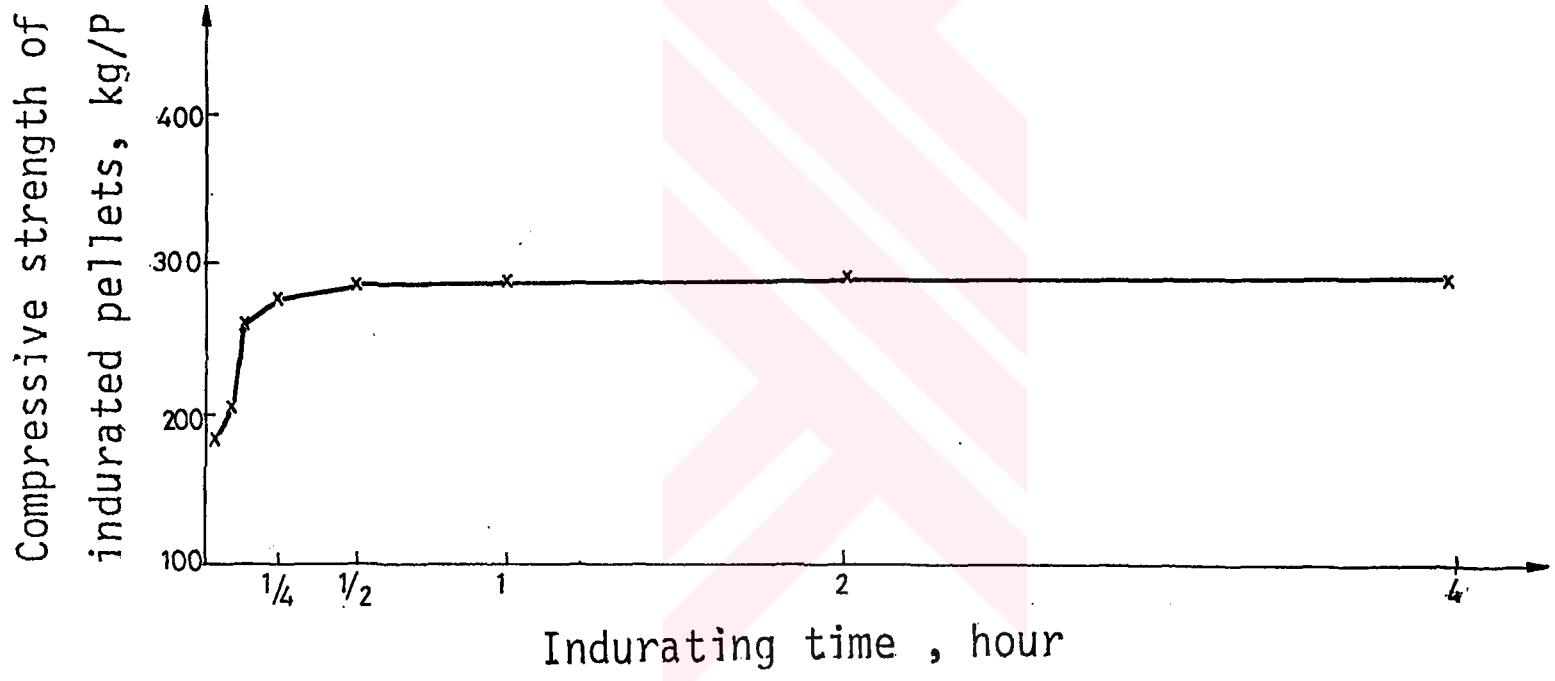


Figure 19. Influence of Indurating Time Over Compression Strength of Indurated Pellets.

not vary with time thereafter. In view of the fact that the strengths attained at the end of 15 and 30 minutes of firing are not significantly different, the optimum induration time was chosen as 15 minutes.

The effect of rate of cooling of pellets fired at 1200°C for 2 hours on their strengths is given in Table 5. The strengths of the pellets are seen from this figure to be very sensitive to cooling rate.

The highest strength was obtained at a cooling rate of ~20°C/minute which was obtained by the following succession of steps: (i)the pellets were removed from the firing furnace kept at 1200°C and immediately placed into furnace kept at 600°C, (ii)the pellets were held in the pot furnace kept at 600°C for 30 minutes, (iii)the pellets were removed from the pot furnace and immediately placed into a drying oven kept at 105°C, (iv)the pellets were held in drying oven kept at 105°C for 10 minutes and then subjected to air at room temperature. The pellet strengths decreased with increased cooling rate and a very low strength of 30 kg/pellet was obtained at a cooling rate of ~1050°C/minute corresponding to quenching in water. Taking into account the practical way of the process and also the results presented in Table 5. ~60°C/minute was chosen as the optimum cooling rate.

4.5. Weight Loss and Porosity of Pellets

Pellets were indurated in two steps. Pellets were first fired at 600°C for 30 minutes and then fired at 1100, 1150, 1200 or 1250°C for different

TABLE 5. Effect of cooling rate on Compression Strength of Indurated Pellets.

Cooling Mode	Compressive Strength of Indurated pellets, kg/P
A	30
B	60
C	115
D	130

A : Pellets indurated at 1200°C for 2 hours were quenched into water.
B : Pellets indurated at 1200°C for 2 hours were cooled in air.
C : Pellets indurated at 1200°C for 2 hours were placed into a pot furnace kept at 600°C, held here for 15 minutes and then cooled in air.
D : Pellets indurated at 1200°C for 2 hours were placed into a pot furnace kept at 600°C, held here for 15 minutes and then placed into an oven kept at 105°C, held here for 10 minutes and finally cooled in air.

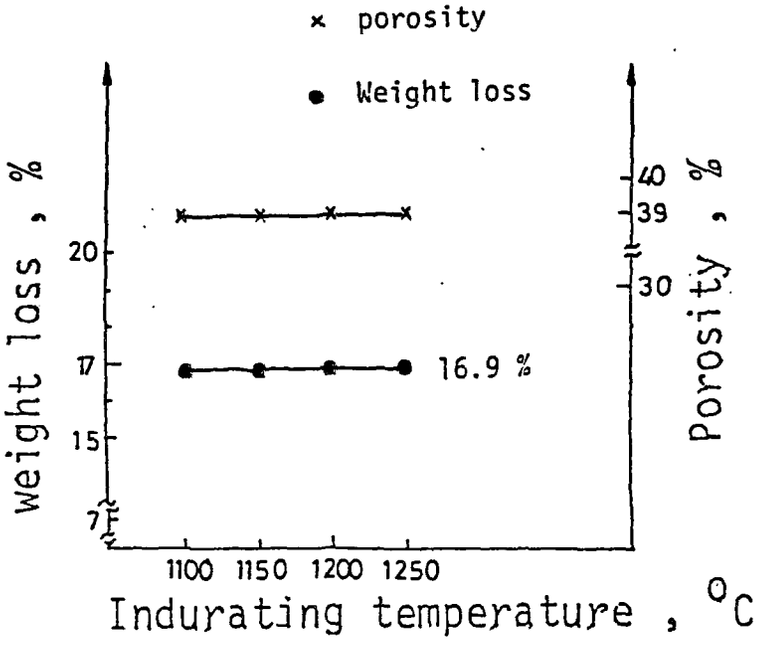


Figure 20. Variation in Weight Loss and Porosity vs Indurating Temperature.

periods of time, weight loss due to calcination was experienced during the firing steps. The weight loss of the pellets fired at 600°C for 30 minutes was ~15%. An additional~2% loss in weight was detected for pellets fired at higher temperatures and this weight loss was independent of firing temperature as shown in Figure 20. The porosities of the pellets were also independent of firing temperature as can be seen from Figure 23.

4.6. Microstructural Examination of the Ore Fine and of the Pellets

The micrograph taken from thin sections of the Hekimhan sideritic iron ore fine is shown in Figure 21. Different colors in the micrograph indicate different crystals as presented below: Brown and Light brown regions: mangano siderite crystals (Siderite contains 12 % $MnCO_3$), light and dark gray regions: calcite and dolomite, black regions: magnetite, yellow and blue regions: silicates.

The main ore minerals are seen from Figure 21 to be mangano siderite. The main gangue components on the otherhand are silicates and small amount of dolomite.

The micrographs taken from the pellets are shown in Figure 22(a-b-c-d-e-f). Different colors in these micrographs indicate different crystals as presented below:

White regions : hematite and magnetite crystals

Light gray regions : Calcium and magnesium ferrites

Dark gray regions : Glassy calcium magnesium silicates.

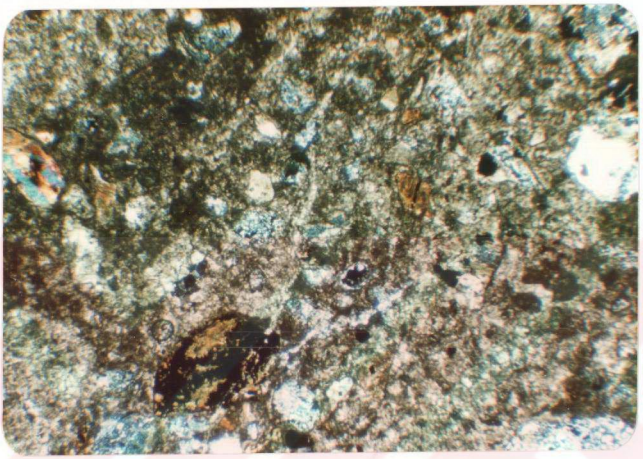


Figure 21. Microphotograph of Thin Section of Hekimhan Sideritic Iron Ore Fine (125 X).
Manganosiderite (Brown, Light brown)
Calcite and dolomite (Light and dark gray)
Magnetite (Black)
Silicates (Yellow and blue)

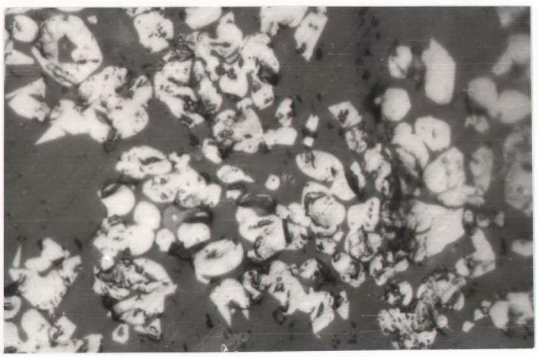


Figure 22a.Microphotograph of Pellets of Hekimhan Sideritic Iron Ore Fine Indurated at 1100°C and Subcooled at 600°C for 15 minutes(400 X).

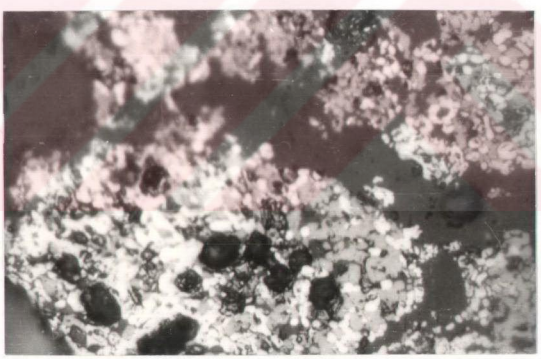


Figure 22b.Microphotograph of Pellet of Hekimhan Sideritic Iron Ore Fine Indurated at 1150°C and Subcooled at 600°C for 15 minutes (400 X).

Hematite and Magnetite (White)
Silicates (Black)
Calcium Ferrites (Gray) , in microphotographs of pellets.

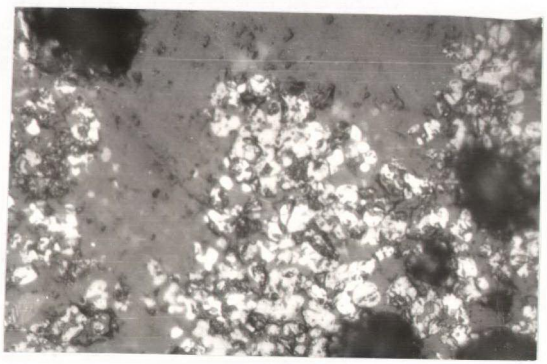


Figure 22c.Microphotograph of Pellet of Hekimhan Siderite Iron ore Fine Indurated at 1200°C and Subcooled at 600°C for 15 Minutes (400 X).

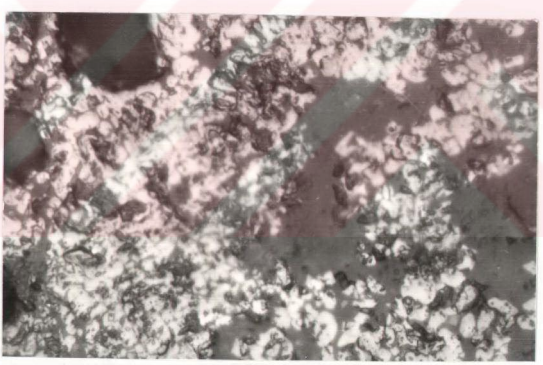


Figure 22d.Microphotograph of Pellet of Hekimhan Sideritic Iron Ore Fine Indurated at 1250°C and Subcooled at 600°C for 15 Minutes (400 X).

Hematite and Magnetite (White)
Silicates (Black)
Calcium Ferrites (Gray) , in microphotographs of pellets.

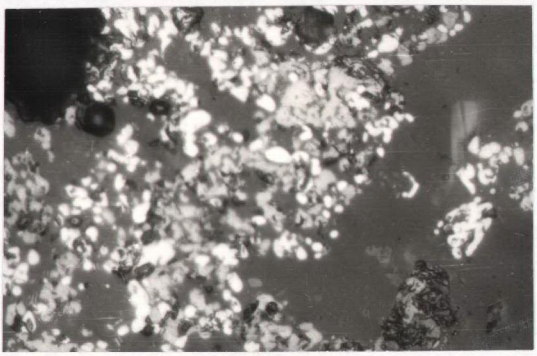


Figure 22e. Microphotograph of Pellet of Hekimhan Sideritic Iron Ore Fine Indurated at 1200°C and Suddenly Cooled in Air (400 X).

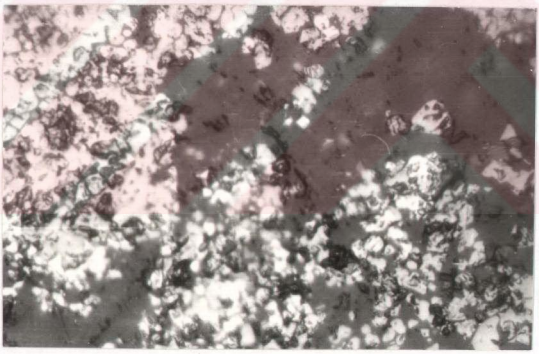


Figure 22f. Microphotograph of Pellet of Hekimhan Sideritic Iron Ore Fine Indurated at 1250°C and Suddenly Cooled in Air (400 X).

Hematite and Magnetite (White)
Silicates (Black)
Calcium Ferrites (Gray) , in microphotographs of pellets.

It is seen from the micrographs given in Figure 22 that the binding medium between the hematite grains is glassy calcium-magnesium silicates. The ferrite occurrence, light gray, observed at 1100°C and 1150°C could not be detected in the micrographs of the pellets indurated at 1200 and 1250°C. The grain size of hematite is seen from Figures 22 a-b-c-d to decrease with increasing induration temperature. Increased cooling rate also decreases the grain size of hematite as can be seen from a comparison of Figure 22c and 22e. Hematite grains are seen to be unassimilated from each other and precipitated as long needle-like crystals when cooling rate is high (Figure 23e as compared with Figure 23c).

4.7. Properties of Pellets Produced from a 50:50 Mixture of Hekimhan Fine and Dumluca Magnetite Fine

Hekimhan siderite fine was mixed with Dumluca magnetite fine in a 50:50 weight ratio. Pellets were then produced from this mixture under conditions mentioned as optimum above.

The green and dry strengths of these pellets are shown in Figure 16 as two shaded rectangular areas. The green strength and dry strength of these pellets were ~2 kg/cm^2 and ~9 kg/cm^2, respectively, as shown in Figure 16. The dry strengths of these pellets are greater than those produced from only Hekimhan fine.

Porosity of these pellets is approximately 30% as shown in Figure 17 in a shaded area, and lower than those produced from only Hekimhan fine. This porosity is sufficient for use in blast furnaces. The compression

strength is greater than the compression strengths of pellets produced from Hekimhan fine only,which is primarily due to lower porosity.

Green and dry strengths of spherical pellets produced from Hekimhan fine are ~1.5 kg/cm^2 and ~4.5 kg/cm^2,respectively which are lower than those of cylindrical pellets produced from Hekimhan fine, as shown in Figure 16. The compression strength of spherical pellets is ~200 kg/P and is sufficient for use in blast furnaces. Porosity of the spherical pellets is ~39% and higher than that of the cylindrical pellets as shown in Figure 17.

5. CONCLUSIONS

The results of this investigation indicate that pellets having sufficient wet and dry strengths can be produced from Hekimhan siderite fine without using any binder. The most important conclusion of this study is that pellets can be produced from uncalcined Hekimhan siderite fine calcination of which takes place during induration heating.

Optimum moisture content of the pelletizing mix has been found as 10%. Optimum induration temperature was determined as 1200^{o}C. It was found that 15 minutes of firing at this temperature was sufficient for the pellets to be indurated. Cooling rate has been found to effect the pellet strength significantly. Optimum cooling rate was determined as 60^{o}C/minute.

Compression strengths of pellets prepared from a pelletizing mix with 72% of the particles were finer than 105 μm under conditions stated as optimum above were larger than 200 kg/pellet. Porosity of these pellets was 39% which, being quite high, is expected to influence the reducibility of the pellets positively.

Hekimhan siderite fine has ~32% Fe in it. The Fe content of the indurated pellets produced from this fine increases to ~42% as a result of calcination but is still too low for use in blast furnaces. Hekimhan siderite fine was mixed in a 1:1 weight ratio with Dumluca magnetite fine to increase the Fe content of the pelletizing mix. It was possible to obtain even stronger pellets from this mixture; the Fe content of these pellets was ~50%.

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