Making and Properties of Mixed Magnetite-Hematite Pellets

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THE paper describes pelletizing trials with increasing additions of hematite fines and firing at various temperatures. With 50 per cent hematite, the strength after firing remains high and reducibility is not changed. The mixed 50 per cent hematite pellets have a strength comparable to that of magnetite pellets.

Aim of research

The pelletizing technique for fines treatment is today a current practice in iron industry. High grade ores of suitable size for direct charging in blast furnace become more and more rare. Techniques of low grade ore treatment are therefore more and more developed, in order to increase iron content to reach limits suitable for use in blast furnace ; that requires sintering of resulting fines.

Beneficiation requires in effect grinding and crushing to separate gangue from the concentrate.

Among different sintering processes, pelletizing[1] has been developed to use very fine ores with high iron content, generally produced by magnetite concentration. Their treatment in usual sintering plants presents sometimes great difficulties and reduces considerably the productivity of the plant.

The magnetite fines are the most suitable for the pelletizing process.

During firing of pellets of magnetite fines an oxidation process to hematite takes place ; this oxidation causes the development of chemical bonds in the pellets, which, after transformation, are very resistant and in consequence their behaviour during the following operations of handling, charging, etc., is very good. The oxidation of magnetite and hematite occurs already at relatively low temperatures. With increase of temperature, hematite grains, as soon as they are formed, grow and bond themselves one another so as to give consistency and solidity to pellets.

During burning of magnetite pellets, a very resistant chemical bond is developed ; it explains indirectly the failure of numerous experiments for production of pellets with satisfactory mechanical properties by hematite fines ; these fines, in the best of cases, could aggregate only by firing at very high temperature.

In order to utilize high grade hematite fines (Venezuela ore type) it is however very interesting to study the possibility of making pellets of suitable strength by mixing with magnetite fines, without necessity of working at too high temperatures, but taking advantage of the transformation of magnetite fines into a product of higher grade of oxidation.

The present research has been carried out to determine the limits within which it is possible to add hematite fines to magnetite concentrates in the pelletizing process.

The aim of the research was also the study of influence of firing temperature on strength of fired and reduced pellets and the measure of their reducibility.

Trial scheme

In order to get some information about the behaviour of pellets produced with hematite and magnetite fines, during firing and reduction process and chiefly about pellets strength after firing, a set of trials has been designed with five types of mixtures which fired at four different temperatures.

The ores used were Venezuela hematite and S. Leone magnetite concentrate.

Five different mixtures have been tested. Compositions are given in Table I.

TABLE I

Symbols and composition of ore mixtures

Symbols	Composition by weight %	
	Venezuela	S. Leone ore
4V	100	—
3V 1S	75	25
2V 2S	50	50
1V 3S	25	75
4S	—	100

Firing has been carried out at four different temperatures for each type of pellets: 850–950–1050–1150°C *with factorial experiment.*

Twenty different types of pellets, different compositions and firing temperature, have thus been prepared. Preparation and firing have been carried out at random.

This experiment of factorial type 5.4., allows, after variance analysis, an objective determination of the effects of mixture and temperature with risk of predeterminate error.[2]

Ore treatment

The S. Leone ore has been drawn from a deposit of known size fines.

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Venezuela ore had a 0–10 mm size, and therefore it has been necessary to grind it, in order to reach a grain size as near as possible to that of S. Leone ore.

After numerous trials, each followed by grain size analysis, we hoped to reach a suitable grain size by this procedure:

Venezuela ore has been screened with a 50 mesh sieve, so that two fractions are obtained: <50 mesh fraction, which is 40 per cent by weight of treated ore; >50 mesh ore, which is 60 per cent by weight of treated ore.

This latter fraction has been crushed in a rod mill for 8 minutes and mixed with the former fraction;

—by mixing the two fractions, an ore fine is obtained with nearly the desired size.

Preparation and firing of pellets

After the above treatment of ore, pellets of various fixed compositions were made. The pelletising has been carried out in a small drum 40 cm in diameter and 20 cm long. Pellets of 26÷30 mm dia. have been selected and then fired in a Silit resistance furnace for heat treatment.

After introduction of the box containing pellets in the cold furnace, the furnace temperature with open doors was slowly raised to 250°C. This temperature was maintained for about 1½ hours in order to allow a steady evaporation of moisture and to avoid cracks and decrepitation.

The temperature was then raised up to the desired value and maintained for three hours. The furnace was then switched off and slow cooling took place.

Breaking test

Testing method: In order to obtain data on pellets strength, compression breaking load test has been used. Its advantage in respect of other tests such as drop and tumbler tests, is that it can be performed on a rather limited number of pellets and can give numerous data useful for treatment comparisons even though their independence is strictly relative.

Since the resulting strengths vary widely according to composition and firing temperature, two different equipments have been used to perform the breaking test.

When the pellets had a strength lower than 100 kg (total load), tests have been performed with a wire tensile testing machine used in compression; pellets with greater strength have been tested with a Brinell hardness tester, without the penetrator ball.

Breaking tests have been performed for each type of pellets.

We wanted to make a number of observations sufficient to compensate variations in mixing (grain-size, moisture, etc.), firing and in pellets diameter of each single type.

We assumed an average diameter of 28 mm; effective deviation $\pm 3\sigma$ from this value was of ± 2 mm.

Results of breaking test: Table II contains mean values of breaking total loads for the different types of pellets. Each value is the average of 30 values.

TABLE II

Total breaking loads (kg) of fired pellets. Average of 30 values.

		Mixtures				
		4S	3V 1S	2V 2S	1V 3S	4S
Temperatures	850	5·11	8·43	7·96	15·52	39·83
	950	11·67	12·50	15·97	28·95	43·53
	1,050	16·75	32·53	58·10	172·73	245·00
	1,150	106·16	158·06	374·50	446·83	564·33

Data dispersion is considerable and usually increases with higher average breaking strength for each type of pellets.

In order to increase effect of distribution obliquity, we have thought it advisable to perform logarithmic transformation of data, as shown in Table III. Values reported in the table are the mean of logarithms of original breaking loads for each type of pellets.

They are not obviously logarithms of means reported in Table II.

Histograms of Fig. 1 report the frequency distribution of single values of breaking tests.

Each histogram is formed by thirty data. The frequencies are graphed as ordinates and the total loads as abscissae on logarithmic scale.

TABLE III

Total breaking loads. Logarithmic transformation.

		Mixtures					Temp. mean	Antilog
		4S	3V 1S	2V 2S	1V 3S	4S		
Temperatures	850	0·660	0·891	0·874	1·178	1·591	1,039	10·94
	950	1·038	1·087	1·178	1·443	1·615	1,272	18·70
	1,050	1·200	1·478	1·837	2·189	2·328	1,806	64·00
	1,150	2·005	2·088	2·557	2·641	2·737	2,406	255·00
Mixtures mean		1,226	1,386	1,612	1,863	2,068	Gen. mean 1,631	
Antilog		16·8	24·3	40·9	73·0	117·0		42·8

A rapid examination of the data contained in Table II shows that compression strength of pellets increases greatly with firing temperature and with increasing content of magnetite ore S. Leone.

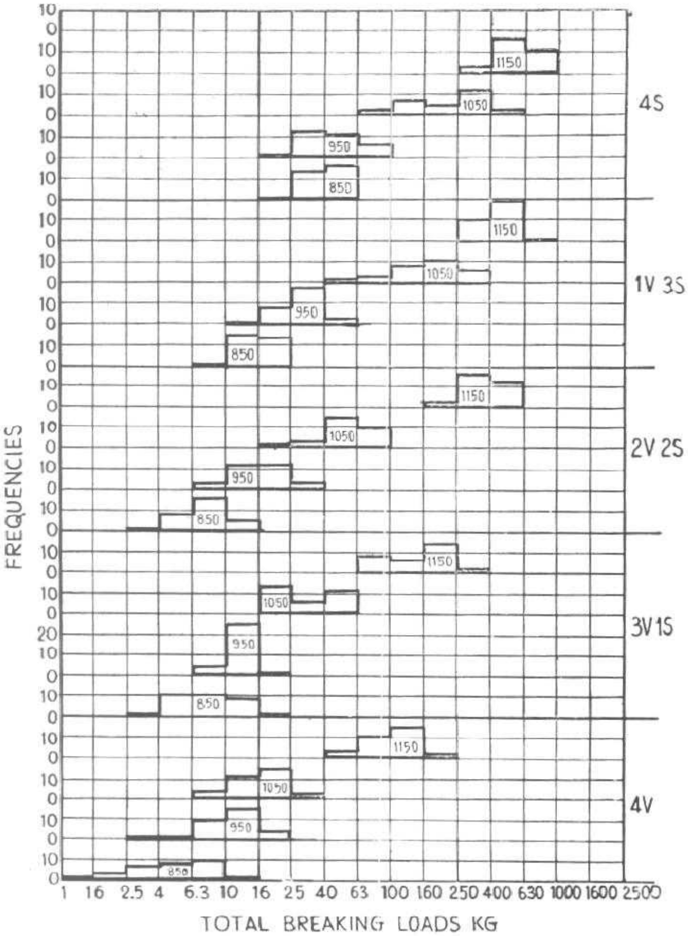

Fig. 1.

Frequency distribution of breaking loads of fired pellets.

The quantitative interpretation is possible with variance analysis on data of Table III; the results are reported in Table IV. Effects of mixing and firing temperature are very considerable.

TABLE IV

Variance analysis.

Source of variation	Degrees of freedom	Σq	Mean squares	F ratio	Significance
Mixture	4	1·876	0·469	24·427	♀ ♀
linear component ...	1	1·876	1·867	97·240	♀ ♀
residual ...	3	0·009	0·003		
Temperature ...	3	5·551	1·850	96·354	♀ ♀
linear component ...	1	5·370	5·370	279·688	♀ ♀
quadratic component	1	0·167	0·167	8·698	♀
residual ...	1	0·014	0·014		
Error ...	12	0·230	0·0192		
Total ...	19	7·657			

From the following analysis of means of different treatments by Duncan[3] three groups of breaking loads, which are significatively different, are obtained:

4 V	low	
3 V	1 S	strength
3 V	1 S	
2 V	2 S	middle
1 V	3 S	strength
1 V	3 S	high
	4 S	strength.

They are ranged according to increasing resistance to breaking.

The influence of increasing temperature is no doubt also considerable. For all temperature increases (except 850 and 950°C, that are indistinguishable) significantly different breaking loads are obtained.

Figs. 2 and 3 show graphically the influence of firing temperature and composition on resistance to breaking; the curvature (quadratic component) of the temperature effect is semi-significative.

With raising of firing temperature, the increase of strength is quadratic.

The points in diagrams of Figs. 2 and 3 are anti-logarithm of values of Table III.

Reducibility test

Testing method: A sample of 500 gr (about 20 pellets) is taken from each type of pellets for measuring the reducibility in hydrogen with an apparatus developed by Istituto Siderurgico "Finsider".[4] At first the furnace was heated in a stream of high purity nitrogen at 850°C, and then reducibility test was performed at this temperature with hydrogen as reducing agent. The rate of hydrogen flow was held at 1,000 litre per hour.

Reduction running has been followed by continuous control of developed water, which is removed by means of absorbers placed on a scalepan.

The result of each reducibility test is expressed as T 90, time required for 90% reduction.

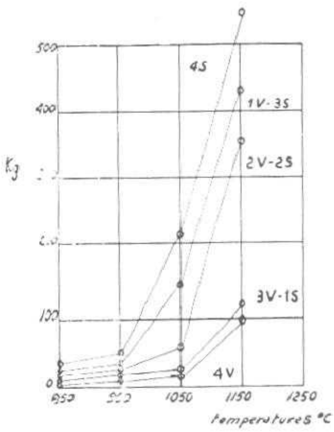

Fig. 2.

Total breaking loads of mixed hematite=magnetite pellets. Curves with mixture of constant composition.

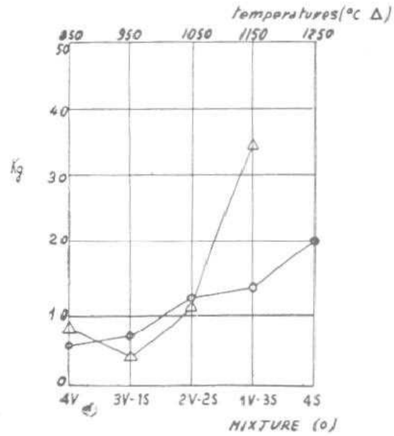

Fig. 3.

Total breaking loads of fired pellets. Means for firing temperature and mixture.

Diagrams of Fig. 4 show two reducibility tests on Venezuela and S. Leone ore pellets.

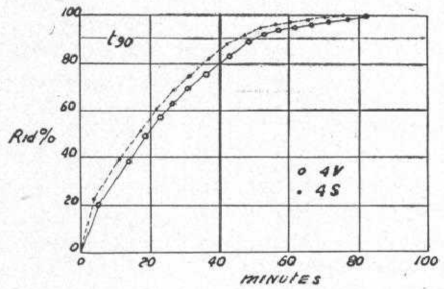

Fig. 4.

Reducibility curves of pellets made from Venezuela and S. Leone ore. Reduction by hydrogen 1,000 l/h. Temperature of test 850°C.

At the end of the test, after slow cooling of the sample in furnace in the stream of high purity nitrogen, the analysis of the produced iron sponge and the determination of reduction grade (expressed as the ratio between metallic Fe and total Fe in iron sponge) are performed.

Metallic Fe has been determined by method of acid etching and hydrogen development.[5]

Results of reducibility test: Values T 90, obtained in the twenty reducibitity tests are given in Table V.

TABLE V.

T90 (Minutes) for 20 tests of reducibility. Reduction temperature 850°C. Gas H_2 1,000 l/h.

		Mixtures					Temper.
		4S	3V 1S	2V 2S	1V 3S	4S	mean.
Temperatures	850	48·7	43·4	45·1	51·6	42·9	46·34
	950	49·8	48·0	53·8	50·5	49·4	50·30
	1·050	52·8	47·4	51·5	53·7	54·3	51·94
	1·150	48·5	53·1	54·7	48·1	56·3	52·14
Mixtures mean		49·95	47·98	51·28	50·98	50·73	General mean 50·18

Variance analysis, reported in Table VI, has been performed on values of Table V. Only the treatment firing temperature, particularly its linear component, is of significance; time required for 90 per cent reduction increases slightly with increase of firing temperature of pellets.

No difference is found in diversity of two Venezuela and S. Leone ores and their mixture (Fig. 5). This is particularly remarkable for the two pure ores: the great porosity of pellets explains the behaviour of these two materials, that are commonly classified as different from this point of view.

TABLE VI

Variance analysis of data of Table V.

Source of variation	Degrees of freedom	Σq	mean squares	F ratio	Significance
Mixture	4	28·17	7·04	0·82	
Temperature	3	108·49	36·16	4·20	♀
linear component ...	1	90·63	90·63	10·53	♀ ♀
residual ...	2	17·86	8·93		
Error ...	12	130·29	8·61		
Total ...	19	266·95			

Breaking test of reduced pellets

Generalities: When the pellets are removed from the furnace, they are often fissured, cracked and many of them are broken and crumbled. Only the pellets of some types are practically sound and tough.

It was interesting to perform breaking tests on reduced pellets, in order to know the average loss of strength and the influence of composition and firing temperatures on residual strength after reduction.

The pellets, after reduction, are fairly plastic and before they show signs of yielding or fracture during compression, they undergo a considerable buckling; this fact is observed only seldom on unreduced pellets.

Results of breaking test on reduced pellets: The mean values (from ten results) of total breaking loads of reduced pellets are reported in Table VII. In reference to strength after firing (general mean 118·23) the strength after reduction is greatly decreased (general mean 17·09).

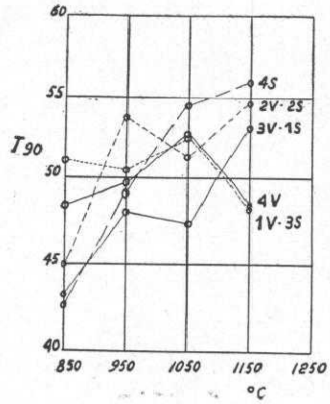

Fig. 5.

Reducibility tests of pellets T90 values for various compositions.

Table VII

Total loads (kg) of reduced pellets. Average of 10 values.

		Mixtures				
		4V	3V 1S	2V 2S	1V 3S	4S
Temperatures	850	4·57	6·30	7·80	12·63	21·80
	950	1·98	3·41	6·48	8·13	6·77
	1050	9·17	7·32	10·08	11·33	24·85
	1150	18·722	3·18	52·66	39·72	64·96

The frequency distribution of total loads is shown in Fig. 6. The great variations of results does not allow an immediate interpretation of experiments with the graphic method; variance analysis of data, after logarithmic transformation (Table VIII) proves that breaking strength after reduction increases with increasing content of magnetite ore and firing temperature (Table IX).

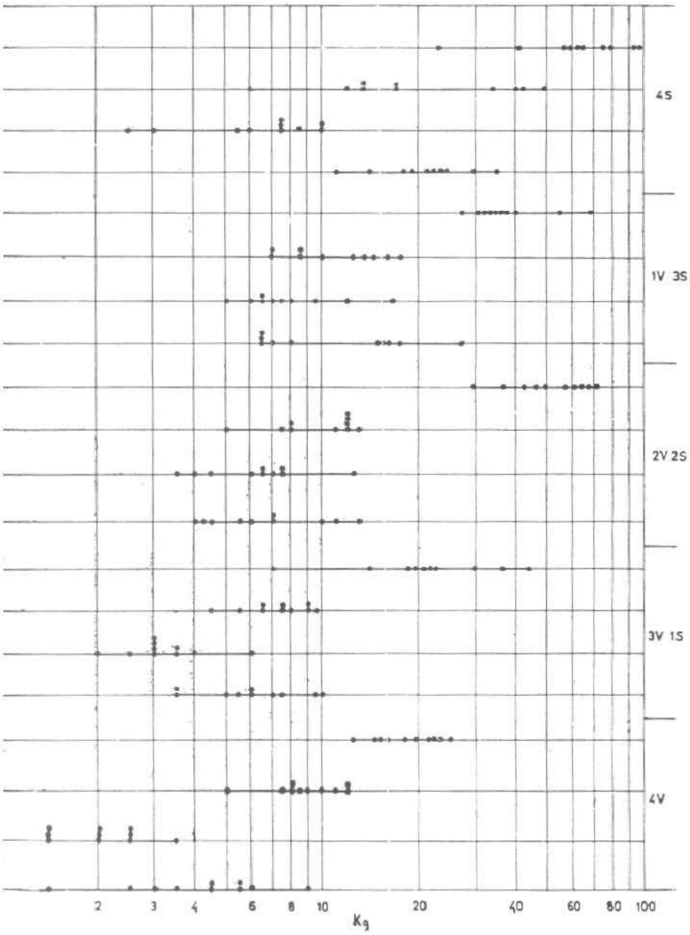

Fig. 6.
Frequency distribution of total loads in breaking tests of reduced pellets.

Mixture range in three groups significantly differ from each other:

4 V	low
3 V	1S strength
3 V	1S middle
2 V	2S strength
2 V	2S
1 V	3S strength
	4S strength

With regard to firing temperature influence, the groups 950, 850-1050 and 1150 are remarkable at 950°C the strength shows a minimum also confirmed by the existence of quadratic component of effect, the interpretation of which is not clear. Figs. 7 and 8 show the strength after reduction with different mixtures and temperatures.

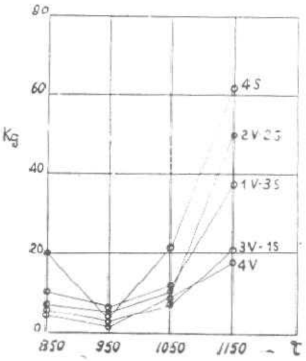

Fig. 7.
Breaking loads of reduced hematite-magnetite pellets. Curves with mixture of constant composition.

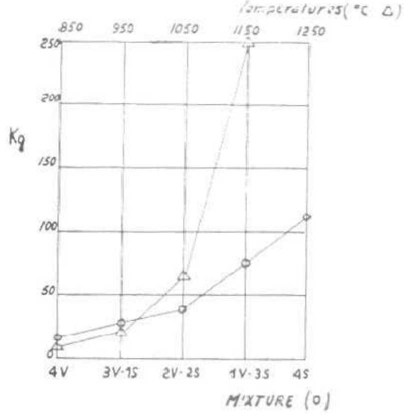

Fig. 8.
Total breaking loads of reduced pellets. Means for temperature and mixture.

Conclusions

Pellets of hematite-magnetite mixtures are made from fines with variable composition of hematite (Venezuela ore) and magnetite (S. Leone ore), using different firing temperatures.

It is known that chemical bond in fired pellets is due to oxidation and so hematites, without additions of other materials, are not suitable for pelletising. Hematite fines however may be added in mixture, without the strength of fired pellets, which is lowered considerably with increasing additions of hematite decreases below technologically allowable limits.

This process is a possible method of utilisation of high grade hematite fines.

High firing temperature increases pellets strength; the influence on reducibility is low, as the reducibility decreases only lightly with firing temperature.

The behaviour of the two pure ores and their

TABLE VIII

Values of breaking loads of reduced pellets. Logarithmic transformation

		Mixtures					Temperature mean.	Antilog
		4S	3V S1	2V S2	1V 3S	4S		
Temperatures	850	0·6140	0·7759	0·8576	1·0462	1·3179	0·9223	8·36
	950	0·2814	0·5134	0·7855	0·8890	0·7918	0·6522	4·49
	1,050	0·9498	0·8534	0·9864	1·0318	1·3235	1·0290	10·69
	1,150	1·2625	1·3176	1·7170	1·5828	1·7906	1·5341	34·20
Mixture mean		0·7769	0·8651	1·0866	1·1374	1·3060	General mean 1·0344	
Antilog		5·98	7·33	12·20	13·70	20·20		Antilog 10·83

TABLE IX

Variance analysis of data of Table VIII

Source of variation	Degrees of freedom	Σq	Mean squares	F ratio	Significance
Mixture ...	4	0·7282	0·1820	14·381	♀ ♀
linear component ...	1	0·7080	0·7080	52·444	♀ ♀
residual ...	3	0·0202	0·0067		
Temperature ...	3	2·0417	0·6806	50·415	♀ ♀
linear component ...	1	1·2233	1·2233	90·615	♀ ♀
quadratic component ...	1	0·1502	0·1502	11·126	♀ ♀
residual ...	1	0·6682	0·6682		
Error ...	12	0·1624	0·0135		
Total ...	19	2·9323			

different mixtures to reducibility test is the same; owing to their high porosity the pellets—also if they are made from magnetite—have a very high reducibility.

After reduction, mechanical properties are considerably decreasing and a favourable increase of temperature and magnetite content in the mixture is also always observed. Up to 50 per cent hematite, the strength is however high.

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