

NAPHTHA REDUCTION OF IRON ORES TO SPONGE IRON IN CONTINUOUS VERTICAL REACTOR

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The development of a process of sponge iron production by naphtha, a petroleum by-product, in a continuous 90 mm I.D. vertical reactor is discussed. The process, the equipment and the operational parameters, the results of the campaigns with various iron ores and pellets are described.

Sized iron ore fines from two deposits were directly reduced by petroleum naphtha to sponge iron at a temperature of about 1000 deg. C, maintaining naphtha injection at different controlled flow rates. The movement of solids in the reactor column was also controlled to obtain predetermined residence times, producing 70—100 kgs of sponge iron per day. Under optimum conditions 98 per cent metallization was obtained.

The results are discussed in the light of efficiency of the reductant and the economics of the process.

AMONGST the various direct reduction processes developed to the commercial stage so far the most successful have been based on gaseous reduction of iron ores. The HyL and the Midrex can be readily mentioned as those which have successfully withstood the test of time and have promoted considerable confidence in the future of the Direct Reduction approach for steel production. Not only have long years of operation helped in overcoming the inevitable teething troubles but the gaseous reductants have brought out some salient advantages of their own. An important amongst them is the purity and cleanliness of the

product which can be directly shipped to melting units without intermediate cleaning such as magnetic separation.

Large number of processes have been developed all over the world using of gaseous reductants produced by reforming natural gas, oil or complete gasification of coal. The type of reactors used for the reduction reactions are retort type, vertical shaft type, fluidized bed type, fluid or fixed bed type or rotary type. The various gas based processes working on commercial or semi commercial scale for sponge iron production in various parts of the world are HyL,

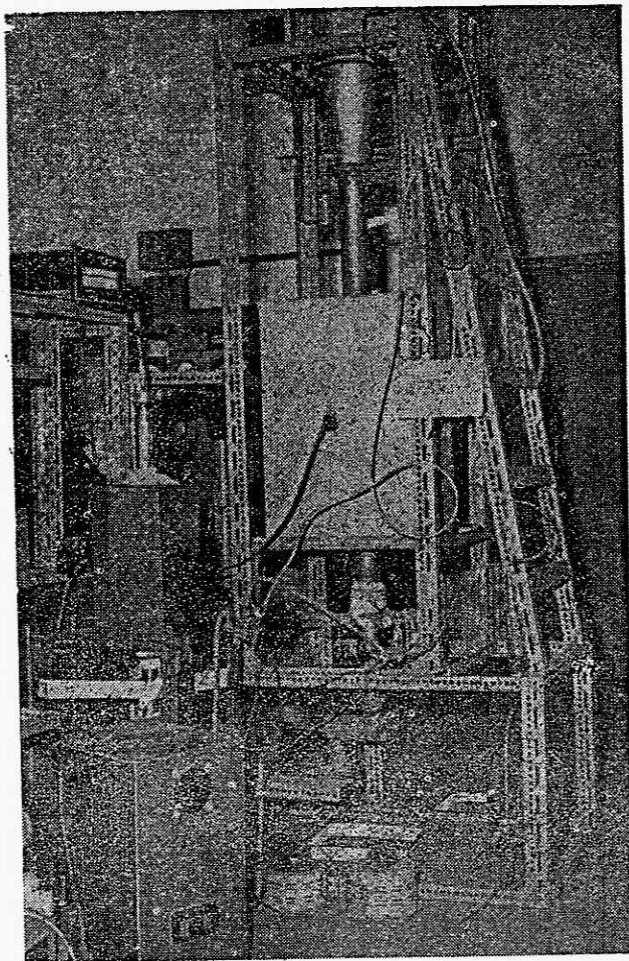


Fig. 1. Continuous vertical reactor for naphtha reduction of iron ores to sponge iron.

The Present Process

Keeping the disadvantages of the previous works in view, it was decided to develop a process which would be more efficient and produce optimum metallization in sponge iron. The first major change was to discard the batch concept and introduce a vertical continuous counter-current system. Reactor diameter, length of reaction zone, iron ore particle size, the rate of descent and rate of naphtha input etc. have been adjusted so as to give the optimum conditions for reduction of iron ores to sponge iron.

(a) Naphtha vaporizer

Total evaporation of naphtha is effected in a vaporizer, specially designed at a temperature of 400-500 deg. C. Naphtha is fed to the vaporiser from a reservoir. The feed is controlled in such a way so as to avoid any unvaporised naphtha remaining in the vaporiser or in the line. All other necessary precautions are taken to avoid condensation of

After each batch of experiment the reduced product was cooled in a non-oxidizing atmosphere. It was observed that this process of evaporation only vaporised the lighter fractions in the beginning. As the reduction rates were very high but after the lighter fractions have been used up the reduction decreased considerably. In this process 40-60 ml of naphtha was required to fully reduce 100 gm of iron ores containing 65-67 per cent iron indicating, thereby, that the efficiency of the reductant by this technique was rather low.

As the naphtha evaporated was directly dependent on the volume of air passed, the composition of air/naphtha vapour mixture could not be controlled beyond certain limits. Moreover, from the study of the end products, it was found out that the efficiency of utilization of the reductants was poor due to insufficient gas/solid contact.

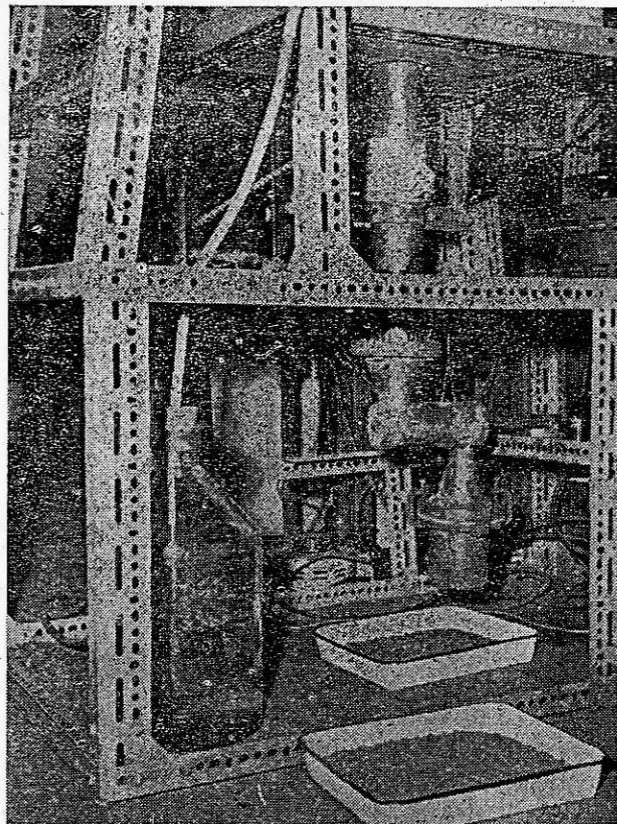


Fig. 2. Discharging arrangement of the reactor.

TABLE—I : Gas based direct reduction processes for sponge iron

Process	Type of equipment	Type of Fuel	Amt/ton of Fe	Energy consumed 100,000 B tu per ton of Fe	Product State	Degree of metallization
Hyl	Static bed	Gaseous reduction (natural gas)	610-710 NM ³ of natural gas	182-240	Sized lumps or pellets	83-90%
ESSO FIOR	Fluidised bed	"	300 kg oil or 900 NM ³ natural gas.	136	Briquettes	92.5%
Purofer	Shaft furnace	"	2570 NM ³ CO+H ₂ per ton Fe, specific gas throughout of the furnace	130	Sized lumps or pellets	upto 95%
Midrex	Shaft furnace	"	Not available	70-130	Sized lumps or pellets	71-95%
Nu-iron	Fluidised bed	Gaseous reduction	300 kg oil or 900 NM ³	140	Briquettes	75%
Armco	Shaft furnace	Gaseous reduction	---	110	Pellets	92%
Wieberg	Shaft furnace	Gaseous reduction	110 kg coal 63 kg oil	300	Sponge	upto 90%

Purofer, FIOR, Midrex and Nu-iron etc. Summarized details of these processes have been given in Table I.

Most of the processes are using the catalytic reforming of natural gas or oil. The reforming gives a gas mixture containing $\text{CO} + \text{H}_2$ in various proportions according to the process of reforming. The efficiency and the amount of gas consumption per ton of Fe mostly depends on the type of reformer used, the efficiency of the catalyst, and the type of reactor suggested for direct reduction.

India is not bestowed with adequate reserves of natural gas. At two places natural gas is available but the total availability is not sufficient enough for installing a sponge iron plant. Also, whatever little gas is available is quite a distance away from the exploitable ore reserves and is associated with many difficulties making any sponge iron plant installation unfeasible. However, the availability of hydrocarbons as petroleum by-product in different forms, still makes it possible to conduct gaseous reduction of iron ores to sponge iron. Naphtha is one of the refinery product which in India can be efficiently used for the sponge iron production. At present, according to Govt. of India's policy, naphtha is primarily earmarked for fertilizer industry. However, apart from naphtha several other hydrocarbons also can be utilised for the production of sponge iron.

Amongst the three processes being actively developed at the National Metallurgical Laboratory, considerable emphasis has been placed on the process using naphtha as a reductant. Extensive trials in our continuous vertical reactor have yielded highly metallized, low carbon, low sulphur sponge iron. Trials have used a number of iron ores from various localities, including Asswan, U.A.R. in form of lumps, fines & pellets.

The technique developed at N.M.L. for producing sponge iron is by directly reducing iron ores fines, lumps or pellets by naphtha in a vertical continuous reactor.

Naphtha — its Properties

Naphtha is the name given to a range of petroleum distillates, volatility of which is such that they could if treated, be used as blending components for gasoline.

Their own Octane ratings are however, far too low for its use in internal combustion engines, but its low sulphur content and freedom from ash, make it ideal for many industrial applications which require clean fuels.

The properties of naphtha are:

The specific gravity at 15°C	- 0.67
Reid vapour pressure	- 12.00
Flash point	-
Distillation boiling points:	
Initial	- 32°C
Final	- 146°C
Sulphur content	- 20-500 ppm
Hydrocarbons analysis:	
Paraffines & naphthenes	- 91%
Aromatics	- 8%
Olefines	- 1%
Carbon : hydrogen ratio	- 5.5 : 1
Calorific value	- 11,275 Kcal/kg.

Naphtha can be easily evaporated to gaseous state because of its low boiling range. For evaporating naphtha any one of the following methods can be utilized:

1. Direct injection of liquid naphtha into a gas stream through pressure jets.
2. Injection of naphtha after vaporising it by using steam as atomising and vaporising agent.
3. Vaporising naphtha in a separate evaporator and passing the vapour into the reactor under pressure.

It should also be noted that when naphtha vapour is injected through pipe lines there is a possibility of condensation of naphtha in them, thereby, choking the system. When naphtha is vaporised in a separate evaporator, the lighter boiling fractions will evaporate first, leaving behind heavy fractions. This will actually change the property and constituents of the gaseous or vapour products from the original properties of naphtha.

However, a total evaporation of naphtha can circumvent the above handicaps.

Development of the Naphtha reduction techniques

In a previous work(1), batch reduction of iron ores with naphtha was tried in a closed circuit in which the reaction products were recirculated through a fuel pump and a naphtha container. The reaction products were thus used as medium for carrying fresh naphtha vapours into the reactor. This was a batch process and entailed difficulties due to excessive loading of the fuel pump as the reaction proceeded.

In another work(2) it was attempted to evaporate naphtha by passing hot air through naphtha kept at a certain temperature. This mixture of naphtha vapour and air was used for reduction in a reactor in which iron ore was kept at a temperature of 1000 deg. C.

naphtha vapour in transit to the reactor. Naphtha vapour from the vaporizer is injected in the reactor under pressure.

(b) Continuous vertical reactor

A vertical reactor made of high oxidation resistant alloy steel has been used with an internal diameter of 90 mm (Fig. 1). Arrangements for continuous charging and discharging (Fig. 2) at the top and bottom have been provided. The entire reactor and its charging-discharging system are made completely leak proof to avoid explosion and pollution hazards. An electrically heated furnace is used to maintain the reaction zone of 500 mm long at a temperature of about 1000 deg. C. By variation of the charging and discharging rates the residence time of the charge in the reaction zone, can be varied between 30–90 minutes. The capacity at the optimum metallization of sponge iron is about 3 to 4 kg. per hour, depending upon the nature of the ore.

(c) The Process

Sized iron ore fines were charged from top of the reactor through the continuous screw feeding arrangement. The naphtha vapour was introduced

through an inlet below the hot zone of the reactor without any catalytic reforming system. As the naphtha vapour ascended the sensible heat of the reduced product coming down in the reactor heated up the naphtha vapour and also cooled down the descending reduced products. Water vapour produced from the hydrogen reduction helped the direct reduction between carbon deposited through cracking of hydrocarbons and iron ore. It also produced more hydrogen to be effectively utilized further up.

Ascending hot reduction products from the reaction zone preheated the descending iron ores thereby further reducing the loss of sensible heat through exhaust gases.

Raw Materials

Considering the diameter of the reactor, permeability of the charge column, gas/solid contact time and surface area of ore, the particle size used in this process was 3-4 mesh B.S.

In view of this, ores employed were taken from fines of two iron ores of different sources. These fines were of relatively poor grades.

Analyses of the two ores are given in Table—II.

TABLE—II
Chemical analyses of two iron ores

Constituents	Iron ore 'B'	Iron ore 'A'
Fe%	59.8	62.6
SiO ₂ %	5.9	1.3
Al ₂ O ₃ %	3.8	3.9
P %	0.035	0.09
S %	0.07	Trace

Other physical properties were also studied such as, bulk density, apparent density, true density, porosity which are given in Table III below:

TABLE—III
Physical properties of iron ores

Property	Iron ore 'A'	Iron ore 'B'
Bulk density	1.81 tons/m ³	1.97 tons/m ³
Apparent density	3.6–3.8 g/cc	3.9–4.2 g/cc
True density	4.5–4.9 g/cc	4.9–5.0 g/cc
Porosity	22.5–27.5%	20–25 %

Results

Metallisation obtained at 1000 deg. C with a naphtha consumption of 100-120 cc/kg of sponge varied from 85-98 per cent depending upon the reducibility of the ore and residence time.

Tables (IV and V) below give results of typical campaigns for two ores of -3+4 mesh size at 1000 deg. C.

The physical properties of the sponge iron produced have given in Table VI.

TABLE IV

Naphtha reduction experiments with iron ore 'A'

Rate of charging in kgs/hour	Naphtha consumed in cc/hour	Rate of discharge of sponge in kgs/hour	% Metallization
3.6	315	2.7	93.8
4.1	330	3.1	96.2
3.3	350	2.5	94.7
3.2	320	2.4	97.0
3.9	350	2.9	98.1
3.5	296	2.6	98.9
4.0	350	3.1	98.9
3.8	350	2.9	86.76
3.3	315	2.6	90.52
3.6	330	2.7	89.2

TABLE V

Naphtha reduction experiments with iron ore 'B'

Rate of charging in kgs/hour	Naphtha consumed in cc/hour	Rate of discharge of sponge in kgs/hour	% Metallization
3.65	367.6	2.79	91.8
3.23	330.0	2.54	83.6
3.69	342.5	2.82	90.8
3.96	332.5	3.02	92.9
2.57	250.5	1.97	91.7
3.19	247.5	2.39	97.7
3.19	310.0	2.40	97.4
3.74	356.0	2.80	99.3
3.55	230.5	2.66	97.8
4.06	284.5	3.05	97.4
3.26	310.5	2.48	93.5
3.13	306.0	2.37	95.1

TABLE-VI

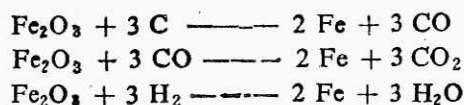
Physical properties of sponge iron

Property	Sponge 'A'	Sponge 'B'
Bulk density	1.6 T/M ³	1.7 T/M ³
Apparent density	3.0-3.5 g/cc	2.8-3.1 g/cc
True density	5.8-6.2 g/cc	5.6-5.8 g/cc
Porosity %	52.3	44.6

The sponge iron produced analysed
It was also observed that there was practically no decrepitation of the ore particles during reduction.

Discussion

A study of the oxygen removal rate during the campaigns (Fig. 3) and also from the efficiency of naphtha utilisation (Fig. 4) it was revealed that the process is a continuous one and the reduction has proceeded almost uniformly throughout. The efficiency of naphtha utilisation is calculated in the following manner. The amount of oxygen that can be removed by the carbon and hydrogen available from a certain quantity of naphtha was calculated on the basis of reduction reactions:



The actual amount of oxygen removed from ore by the same quantity of naphtha is determined from the chemical analyses of the sponge and the ore. Hence the efficiency was calculated by the equation:

$$\text{Efficiency of naphtha utilisation} = \frac{\text{Actual amt. of O}_2 \text{ removed}}{\text{Amt. of O}_2 \text{ that could be removed by the same quantity of naphtha}}$$

It was found from the analyses of the out-let gases that the utilisation of reductants conforms the efficiency calculated by the above equation.

Sponge iron produced was analysed for total iron, metallic iron, carbon, sulphur etc. From metallic iron and the total iron of the sponge iron the percentage metallisation was calculated as under.

$$\text{Percentage Metallisation} = \frac{\text{Percentage of Femet in sponge}}{\text{Percentage of Fe}_{\text{tot}} \text{ in sponge}}$$

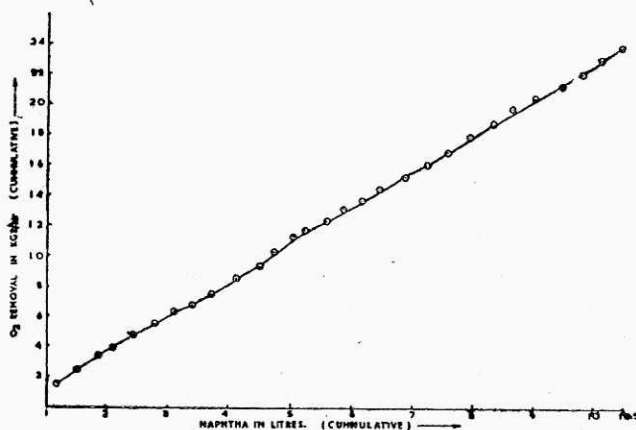


FIG. 3, SHOWING THE PROGRESSIVE OXYGEN REMOVAL WITH NAPHTHA CONSUMPTION.

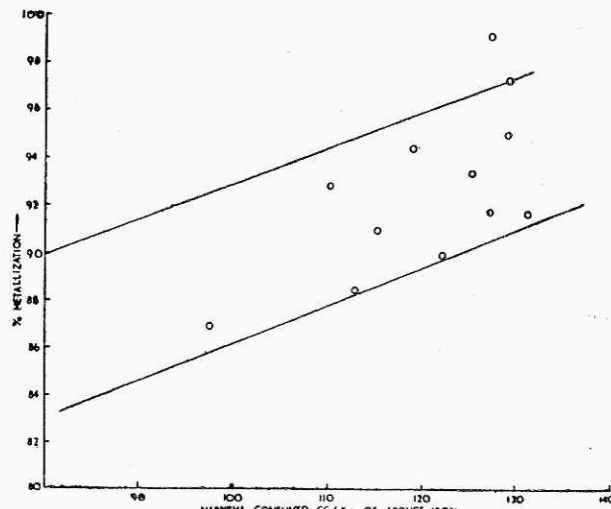


FIG. 4 TRENDS IN PERCENTAGE METALLIZATION WITH NAPHTHA CONSUMPTION

By maintaining optimum conditions carbon can be kept with limits of 0.2—0.4 per cent at a metallisation of over 95 per cent.

As observed from Fig. 3 there is a progressive linear increase of oxygen removal with the naphtha consumed. It also infers that there is a uniformity and continuity of the reduction rate indicating successful operation of the naphtha reduction technique of iron ores for sponge iron manufacture.

Fig. 4 indicates the trend in the increase in percentage metallisation with progressive increase in naphtha consumption per kg. of sponge iron upto certain optimum levels with conditions maintained during the campaigns.

Fig. 5 gives the variation in the efficiency of naphtha with time during the campaign. Though, the variation of 5—8 per cent has been observed during the campaign but this is quite obvious for such vertical continuous reactors. High efficiency of naphtha utilisation is also another indicator of the success of the technique of sponge iron production by naphtha.

Fig. 6, shows the two photomicrographs taken from reduced sponge iron particles. It is clearly revealed that wustite has almost been converted to iron and that this is the advanced stage of reduction.

As discussed in the text, the process is successful with a novelty of not using any catalyst for reforming of naphtha and at the same time maintaining high efficiency of naphtha utilisation at the high-degree of metallisation of sponge iron. Also, naphtha does not have any solid or liquid residue in the sponge, thereby, giving an added advantage of eliminating the intermediate step of separation of sponge from char etc. This may, also result in minimising the heat requirements due to lesser gangue charged in the electric furnace with the sponge during its conversion to steel

Although the campaigns illustrated here refer to medium grade ores, the process has been tried out successfully with richer ores also. It is of course obvious that the value of the sponge for melting into steel will also depend upon its freedom from oxide impurities.

Process utilisation

In view of the present inadequacy of the supplies of Naphtha and the ever increasing requirements of the fertilisers in the country a national policy has been in operation which allows overwhelming allocation of naphtha for fertilizer industry. This may handicap the commercial exploitation of the naphtha process descri-

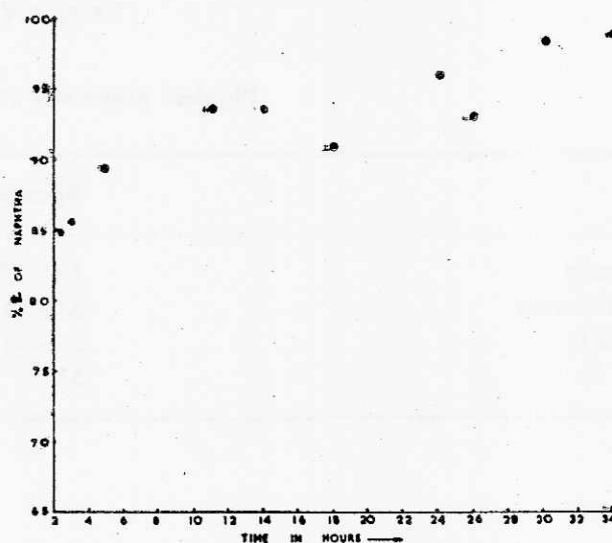


FIG. 5. SHOWING THE GRADUAL INCREASE IN THE NAPHTHA EFFICIENCY WITH THE PROGRESS OF A CAMPAIGN.

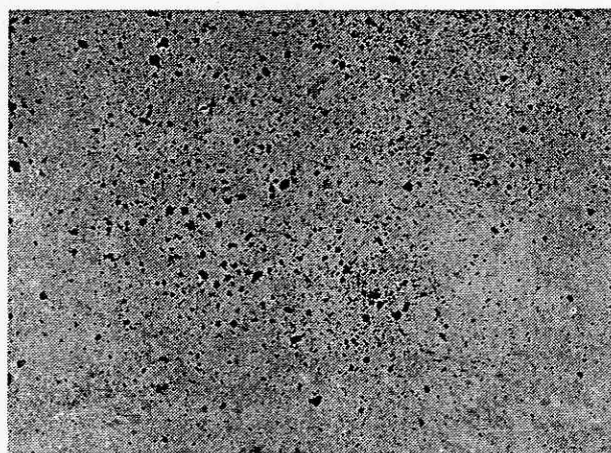
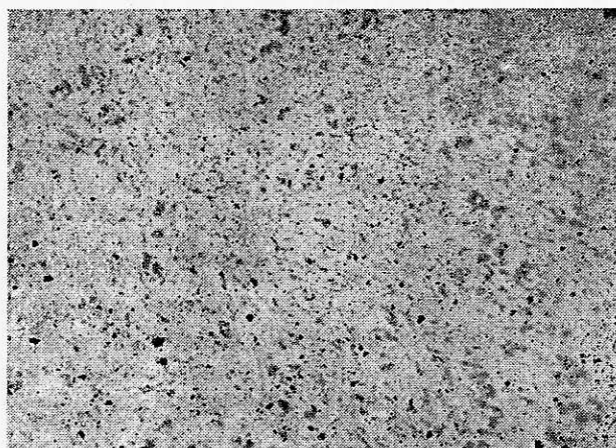


Fig. 6. (a) & (b). Showing advanced stage of reduction with metallic iron phase (white) grains and very faint lines of iron oxide phase (light grey), reflection illumination x 200.

bed herein though this was not the situation at the time of initiation of this study.

We are however hopeful of the following possibilities for the exploitation of this process:

1. Possibility of obtaining naphtha for commercial exploitation of this process in the near future is not really that bleak, as to give up development work on the process.
2. It is quite probable that naphtha may be available in surplus quantities after meeting the demands of the fertilizer industries in future.
3. Coastal plants near Mangalore, Goa or Paradip ports may be installed based on this process taking advantage of Kudremukh, Goa, Barajamda and Kiriburu iron ore deposits in the near vicinity of the ports. It will be worthwhile even to import naphtha at these ports to convert the present exports of iron ores into exports of sponge iron at a great savings. Such a proposal will be attractive to many countries which are traditionally buying ores from us or those countries which want to establish mini-steel plants as in the Middle-East.
4. Last but not the least the technology can be exported to the developing countries of naphtha abundance which are very keen to set up their own mini-steel plants.

DISCUSSION

Mr. A. Chakravorty (SICOM, Bombay): Prof. Altekhar was in favour of using Naphtha as a source of reductant to make sponge iron. It was clearly mentioned by various authors that the availability of Naphtha in India is doubtful. Secondly, the total quantum of Naphtha as might be required to reduce one ton of iron ore to sponge iron is of high tune. If we consider as Naphtha as the reductant, for sponge iron in India; then the question will come what will be the cost of production. It is well known that the scope of using sponge iron to make steel either through electric arc

furnace or using along with hot metal in L.D. is basically the economical condition. If the cost of the sponge iron is not low and doesn't compensate for the additional operating cost, the commercial use of the sponge iron in our country is not far bright.

My desire was to know whether any techno-economical work has been done to find out upto what extent we can pay for this sponge iron without increasing the limited cost of the steel. Obviously, the cost of various materials selected and the fixed expenditure will play the major role. It was pointed out during the discussion that the capital expenditure for a sponge iron complex of a capacity of 1,00,000 tonnes will be around Rs. 900 per annual ton. Considering this as a capital expenditure, fixed expenditure alone will be at the tune of 135 tonnes per ton of production; provided it is 100 per cent capacity utilisation. If the utilisation is lowered, his fixed expenditure will be obviously more. Therefore, we have to work out the cost of production for such a manner that a cost of the sponge iron is lower.

Whether it is possible to have low cost of sponge iron by using Naphtha, or not, considering that Naphtha is available in India, what is the cost of production using Naphtha for sponge iron, if it is being practised in the advanced countries?

Acknowledgements

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Reference

1. B. K. Saxena et. al: Trans. Indian Institute of Metals, Vol. 20, Sept. 1967, p. 145-147.
2. S. R. Joti et. al: Trans. Indian Institute of Metals, Dec. 1969, p. 30-34.