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EUROPEAN PATENT APPLICATION

21 Application number: 79300171.0

51 Int. Cl.2: **C 22 B 1/243**

22 Date of filing: 02.02.79

30 Priority: 03.02.78 JP 10508/78

43 Date of publication of application: 22.08.79
Bulletin 79/17

84 Designated Contracting States: DE FR GB SE

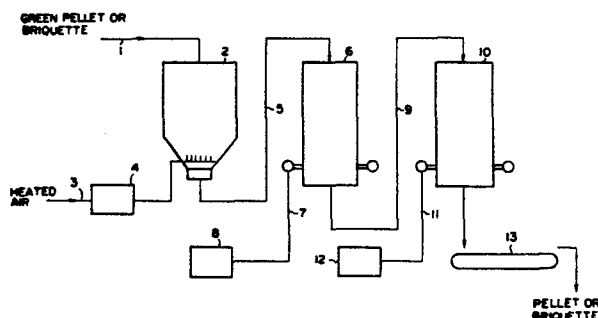
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54 A method of producing cold agglomerates for use in iron making.

57 A method of producing cold agglomerates for use in iron making including the steps of preparing a homogenized mixture of powdered or granular iron-containing material, a hydraulic bonding agent and optionally other additives, adding water to said mixture, homogenizing the resulting mixture, mechanically forming the resulting homogenized mixture into agglomerates, and curing the resulting green agglomerates. In order to produce continuously agglomerates of high strength in a relatively short time, with high operational efficiency and safely without the use of high pressure steam, said hydraulic bonding agent is added in an amount within the range from 3 to 10 wt. parts per 100 wt. parts of said iron-containing material, and the curing is performed by the consecutive steps of allowing the green agglomerates to stand in vessel (2) at ambient temperature up to 60°C and in an atmosphere of normal humidity or from 80 to 100% relative humidity during not more than 3 days, steam treating the resulting agglomerates in steam heating unit (6) at a temperature from 90 to 100°C and then air heating the steam treated agglomerates in heating unit (10) at a temperature from 100 to 500°C.



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A Method of Producing Cold Agglomerates for use in
Iron Making

5 This invention relates to a method of producing cold or unburned agglomerates used for iron making, from pulverized or fine iron ores or other iron containing materials.

10 It is known in the art to agglomerate fine ores by sintering, pelletizing or briquetting. These known methods are usually accompanied by atmospheric and other pollution problems because of the calcinating step in such methods which is carried out at an elevated temperature over 1,100°C.

15 Recently, in order to avoid such environmental problems attention has been paid to a method of cold briquetting in which iron ores are mixed with a hydraulic bonding agent, such as portland cement with water, and formed under high pressures. The resulting green briquettes are allowed to stand until the desired hardness of the briquettes is attained. However, this
20 known method involves a prolonged curing time and therefore does not lend itself to an efficient productivity of cold agglomerates.

25 In order to shorten the curing time in the cold agglomerating process, it has been proposed in the United States Patent 3,676,104 to cure the mixture by subjecting it to steam treatment at high temperatures within an autoclave. This method is not desirable

because of lowered productivity and high production costs on account of the batch type process and it needs special care for safety against the high pressures developed in the autoclave.

5 It is also known from the United States Patent 3,214,263 to produce cold briquettes by the steps of briquetting iron ores by using granulated slag of blast furnace and slaked lime as binder, drying the resulting green briquettes in air for several days, steaming the
10 resulting product at 120°C for 6 to 8 hours and again drying the steamed briquettes in the air. However, as the steam used is at 120°C and therefore unsaturated or at a high pressure so that the productivity of the
15 subjecting process is low and moreover the strength of the resulting briquettes leaves much to be desired.

 It is also known from the United States Patent 4,049,435 to prepare cold lumps or agglomerates by
20 subjecting formed green lumps directly to a heat humidity treatment at 70 - 100% relative humidity and a temperature of 60 to 250°C and then to a two-step heat treatment at
25 70 - 100°C and 100 to 900°C for drying the thus treated lumps. This method is again not satisfactory since a pressure vessel is required for maintaining a temperature of over 100°C at more than 70% relative humidity, thus
30 resulting in increased production costs. Moreover, the strength of the resulting cold lumps again leaves much to be desired.

 The present invention is intended to provide methods of producing cold agglomerates used for iron making,
30 whereby cold pellets or briquettes of high strength may be obtained continuously with a high operational efficiency. It also has the advantage of providing methods of producing cold agglomerates used for iron making, which
35 can be practiced continuously with high safety without the necessity of using high pressure steam.

 A method according to the invention of producing

cold agglomerates for use in iron making is characterized in that to 100 weight parts of fine or pulverized iron containing material are added 3 to 10 weight parts of a hydraulic bonding agent and optionally a desired amount of other additives, the resulting mixture is added with water and homogenized, the so homogenized mixture is then mechanically processed into green briquettes or pellets, and the resulting briquettes or pellets are cured by the steps of

- a) Setting at room temperature during not more than 3 days;
- b) Steam treating at about 100°C; and
- c) Air heating at a temperature 100°C to 500°C.

According to a preferred embodiment of the present invention, the step a) may be carried out at an ambient temperature up to 60°C in an atmosphere of from 80 to 100% relative humidity during not more than 3 days, and the step b) may be carried out at a temperature from 90°C to 100°C.

With this embodiment of the present invention, cold agglomerates of a high compressive strength may be obtained within a relatively short time.

The following is a description, by way of example, of specific methods of producing agglomerates for use in iron making, reference being made to the accompanying drawings, in which:-

Figures 1 to 3 are graphic views illustrating the compressive strength of cold agglomerates produced by methods according to the present invention, as plotted against the operating conditions used in the steps of setting, steam treating and heating; and

Figure 4 is a schematic block diagram showing the curing steps according to a preferred embodiment of the present invention.

Examples of iron containing materials which can be used as starting material for a process according to

the invention are iron sand, iron dusts produced and recovered in iron making plants and fine iron ores. However, pulverized iron ores or fines are most preferred as starting materials. In the following description of processes according to the invention fine iron ores are used as the starting material.

In a process according to the invention, the fine iron ore is mixed with a hydraulic bonding agent and optionally with other known additives, then homogenized with water, and the resulting homogenized product is mechanically formed into green briquettes or pellets. A detailed description of the method of forming such green briquettes or pellets will not be made here because any methods known in the art for formation of such pellets may be used.

A method according to the invention enables an improved compressive strength of the agglomerates to be achieved notwithstanding the fact that the amount of the hydraulic bonding agent is kept to lower than 10 wt. percent related to the weight of the fine iron ore. It should be noted that an increase in the amount of the hydraulic bonding agent in such cold agglomerates may give rise to increased slag formation during iron making. On the other hand, the compressive strength of the agglomerates will not be sufficient if the hydraulic bonding agent is present in amounts less than 3 wt. percent related to the weight of fine iron ore. Typical examples of hydraulic bonding agents are agents such as pulverized portland cement clinkers and blast furnace slags. The marketed portland cement such as standard portland cement and rapid-hardening portland cement, belongs to a category of said crushed portland cement clinker. When the blast furnace slags are used as hydraulic bonding medium, use is made of 2 to 12 wt. percent of slaked lime related to the weight of slags used as accelerator for the hardening reaction. As optionally used additives,

modulators for adjusting the basicity of the slags such as converter slags and slaked lime, and improvers for the properties of the agglomerates may be mentioned besides reaction accelerators as stated above.

5 The fine iron ores are mixed uniformly with bonding agents and the optional additives and the resulting mixture is added to and further mixed with 0.5 to 20 wt. percent of water. The resulting product is formed into green briquettes or pellets. Usually, the green
10 pellets are formed by rolling, while the green briquettes are formed by compression moulding using a suitable mould.

 The resulting green pellets or briquettes are subjected to a curing process which represents the most
15 important feature of the present invention and comprises setting, steam treating and heating steps. The present inventors have found that the total curing time can be advantageously reduced by the curing process of green briquettes or pellets which comprises these three steps
20 under specific conditions.

 The present inventors have also made investigations into the effects of the individual steps of setting, steam treating and heating on curing and have found that
25 different reactions take place for each of these steps and act in concert to improve the overall curing effect.

 Thus, during setting, there occurs mainly an accelerated formation of nuclei of a hydrated substance due to promotion of the reaction of hydration. Such reaction is also promoted during steam treating thus
30 resulting mainly in accelerated growth of the hydrated substance. During heating, some reaction other than the reaction of hydration of the hydrated substance occurs to impart strength to the resulting product. However, excess heating leads to a reduced strength.

35 The present invention has been completed on the basis of the foregoing knowledge and a series of

experiments which are described below. The inventive method will now be described for each of the individual steps, that is, the steps of setting, steam heating and heating.

5 In the step of heating, the pelleted or briquetted material is allowed to stand at ambient temperature for less than three days, or within an atmosphere of relative humidity of 80 to 100% for less than three days at an ambient temperature up to 60°C.

10 An experiment was carried out for determining the effects of the setting conditions on the properties of the product briquettes. The results of the experiment are shown in Figure 1. The starting green briquettes, which were used in this experiment and in the experiments
15 on steam treating and heating to be later described, were prepared as follows. 96 weight parts of goldsworthy ores (I) having the chemical composition as shown in Table 1 and the grain size distribution as shown in
20 Table 2, and 4 weight parts of portland cement clinkers were mixed uniformly, and 10 weight parts of water were added to the resulting mixture. This mixture was
25 homogenized and mechanically worked into green briquettes of 18.1 mm diameter by a briquetting device. The weight of each briquette amounted to about 10 g.

Table I Chemical composition of ores and additives

	T.Fe	FeO	SiO ₂	Al ₂ O ₃	CaO	MgO	Ig- loss
Ores, I(Goldswarlay)	62.6	0.6	7.1	1.4	tr.	0.3	-
Ores II(Goldswathy)	63.8	0.1	4.4	1.3	"	tr.	-
Ores (MBR)	66.9	0.3	1.1	0.1	0.1	0.4	-
Poltland cement clinker	2.6	-	22.0	5.2	65.1	1.3	-
Granulated slag of blast furnace	0.5	-	36.3	14.2	39.9	6.9	-
Calcium carbonate	1.7	-	0.5	tr.	42.6	0.6	42.5
Slaked lime	0.1	-	0.4	0.3	55.9	0.7	24.6
Light burned dolomite	0.1	-	0.5	0.3	52.6	25.6	7.3

(in wt. %)

Table 2 Grain size distribution of ores and additives

grain size (μm)	1000	500	250	105	74	44	-44
Ores I(Goldswarlay)	1.6	2.3	6.0	7.2	12.3	22.7	47.9
Ores I(Goldswathy)	"	"	"	"	"	"	"
Ores (MBR)	0.6	0.1	0.4	1.4	2.8	20.0	74.7
Poltland cement clinker						2.1	97.9
Granulated slag of blast furnace				0.8	0.4	9.0	89.8
Calcium carbonate							100.0
Slaked lime				3.0	1.2	3.7	92.1
Light burned dolomite				35.0	24.5	22.5	18.0

(in wt. %)

The green briquettes thus obtained were maintained in an atmosphere at 15°C with 70% relative humidity, at 15°C with 100% relative humidity, and at 30, 50 and 70°C with 100% relative humidity, for determining the relation
5 between the treating time duration and the compressive strength of the ultimate briquettes. In these test runs, the subsequent steps of steam treating and heating were carried out in steam at 100°C at atmospheric pressure for three hours and in air at 350°C for 1 hour, respectively.
10 The results of these test runs are shown in Figure 1. It can be seen from Figure 1 that the strength of the ultimate briquettes prepared without passing through the setting step is no more than about 100 kg/cm², whereas that of the ultimate briquettes obtained by passing
15 through the setting step may be increased markedly. It is also seen from Figure 1 that the strength of the ultimate briquettes is slightly increased for the setting time of over three days, if the setting temperature of less than about 60°C was used. However, the increase in
20 the strength is that small. With the setting temperature over 60°C, an increase in the compressive strength may be attained for a shorter setting time, however, the strength starts to decrease within three days with lapse of the setting time. In the present invention, the upper limit of the
25 setting time has been selected to be three days, in view of the object of the invention of producing briquettes or pellets continuously within a relatively short time so as to be usable as starting material for iron making. The relative humidity of the setting atmosphere should
30 be increased for preventing evaporation of moisture added as water to the ore and clinker mixture. This may be attained effectively by using a relative humidity over 80%.

The step of steam treating is conducted in steam at
35 90 to 100°C and atmospheric pressure. Figure 2 shows the results of an experiment conducted for determining

the effects of the treating temperature and treating time duration on the property of the ultimate briquettes.

The starting green briquettes as used in this experiment were the same as in the preceding experiment for setting. The green briquettes were set for 1 day at 50°C and 100% relative humidity, treated in steam at 100°C atmospheric pressure for various treating time intervals and heated in air at 300°C for 1 hour.

As seen from Figure 2, the compressive strength of the ultimate briquettes which have not undergone steam treating is that low, whereas that of the ultimate briquettes which have undergone steam heating is increased markedly. As the increase in compressive strength for steam treating in excess of 5 hours is rather smaller, the duration of steam treating should be less than 5 hours, and the steam treating of 1 to 5 hours is preferred. As the steam treating temperature of more than 100°C cannot be maintained under an atmospheric pressure, the upper limit of the steam treating temperature is selected to 100°C. On the other hand, the steam treating temperature of more than 90°C gives good results at a sufficient curing velocity. Thus, the lower limit for steam treating is selected to 90°C.

The heating step is carried out in air at 100°C to 500°C. Figure 3 shows the result of an experiment conducted for determining the effects of various treating conditions on the strength of the ultimate briquettes.

The starting green briquettes as used in the preceding experiments for setting and heat treating were used in the present experiment. These briquettes were subjected to setting at 50°C with 100% relative humidity for 48 hours, then to steam treatment in steam kept at 100°C and atmospheric pressure for 5 hours, and finally to heating under various heating temperatures and heating time intervals.

As seen from Figure 3, those briquettes which have

not undergone any heating, viz, those with zero heating time have a compressive strength of no more than 280 kg/cm², whereas heating imparts sufficient strength to the briquettes to enable them to be used as furnace burdens. Further, as shown in Figure 3, with increase of the heating temperature, the strength will increase more abruptly for a lesser treating time interval, however, there occurs a gradual decrease in strength with the lapse of time. Therefore, the heating time should be selected appropriately according to various heating temperatures.

The results of the experiments shown in and described with reference to Figures 1 through 3 should not be construed as showing all possible effects of setting, steam treating and heating steps on the strength of ultimate products that may be realized in practical production of pellets or briquettes. Each step interacts and affects the compressive strength of the ultimate product. Thus the operating conditions for one step may be varied with changes in those for the other step and with use of different binders or bonding agents. Therefore, the operating parameters for setting, steam treating and heating should be selected more broadly than those considered to be optimum from Figures 1 through 3.

According to the present invention, a set of optimum operating parameters can be conveniently selected under the operating conditions of setting, steam treating and heating as described above. For instance, ultimate agglomerates with a practically sufficient compressive strength can be produced in about 3 days by selecting the time intervals for setting, steam treating and heating 3 days, five hours and 3 hours respectively. However, according to the invention, ultimate agglomerates with practically sufficient compressive strength can be produced in less than 3 days

treating time, and a set of desired operating parameters may be selected depending on the required treating time and in accordance with the operating conditions for the three steps as described above. It is to be noted that, with conventional curing, about one month of curing time will be required for producing the ultimate product with the compressive strength comparable to that obtained by the present invention.

The curing method of the present invention has been described in the above with reference to curing of briquettes. It has been confirmed that substantially similar results may likewise be realized with pellets. The compressive strength of the ultimate pellets obtained by the present method amounts to about 150 to 230 kg per pellet, while that of the ultimate briquettes obtained by the invention amounts to about 200 to 700 kg/cm². Both of these values are sufficient for practical application.

Reference is now made to Figure 4 illustrating the respective steps of the present method. It is to be noted that formation of green pellets or briquettes and sorting or other steps therefor prior to transfer into the present steps may be performed as in the conventional practice and therefore these operations have been omitted from Figure 4.

The green pellets or briquettes which have passed through the above preliminary steps are supplied into a bin 2 via line 1. The bin 2 is a device for setting and supplied by injection via line 3 with air saturated with steam generated in device 4 at certain temperature.

After retention in the bin 2 for a predetermined time, the pellets or briquettes are delivered to a steam treating unit 6 via line 5. The pellets or briquettes descend by gravity through the unit 6 and are discharged therefrom after retention of a predetermined time. A steam inlet 7 is connected to the bottom of the unit 6.

Saturated steam from generator 8 at atmospheric pressure is injected into the unit 6 to flow through the pellets or briquettes accommodated therein and be discharged through an upper opening of the unit 6.

5 After retention for prescribed time in the unit 6, the pellets or briquettes are supplied via line 9 into a heating unit 10. The unit 10 is similar to the unit 6 and has a hot air inlet 11 from a hot air generator 12 connected thereto in place of the steam inlet 7. The
10 pellets or briquettes are heated by the hot air to a predetermined temperature in the course of gradual descent through the unit and, after a retention for a prescribed duration, are discharged through a lower discharge opening onto a conveyor 13 to be thereby transported to a storing
15 place.

 Thus the inventive method can be practiced continuously by a simple device without using high pressure steam. According to the present invention, not only the operating efficiency may be improved, but the costs
20 of various equipment may be reduced markedly.

 The present invention will now be described by referring to numerical examples.

Example 1

Preparation of ultimate briquettes

Goldsworthy ores (II) and portland cement clinkers were used respectively as ores and binders as shown in
5 Tables 1 and 2. 96 weight percents of Goldsworthy ores (II) and 4 weight percents of portland cement clinkers were mixed and to the resulting mixture was added water in an amount corresponding to 10 weight percents based on the weight of the ore cement clinker mixture. The
10 resulting mixture was then homogenized and mechanically worked under pressure into briquettes each weighing 10 g and 18.1 mm in diameter.

The green briquettes thus obtained were allowed to stand for 1 day at ambient temperature and then subjected
15 to a steam treating for 5 hours using 100°C steam and to heating at 350°C for 1 hour. The briquettes thus obtained showed a high compressive strength of 280 kg/cm² inspite of the small amount of the binder present in the starting mixture. The briquettes thus obtained were
20 subjected to a reduction test prescribed in JIS M 8713. The compressive strength of these briquettes following the test amounted to 58.6 kg/cm² and the swelling of these briquettes amounted only to 5.3%, showing that these briquettes may be safely used as furnace burden
25 material.

Examples 2 and 3

Preparation of ultimate pellets

Two pellet samples (CBP No. 1 and CBP No. 2) were
30 prepared. In the first sample or CBP No. 1, portland cement clinker and calcium carbonate were used respectively as binder or bonding medium and basicity moderator. In the second sample or CBP No. 2, granulated
slag of blast furnace were used as main binder, whereas
35 slaked lime was used as reaction accelerator and basicity modulator and light burned dolomite as moderator for

improving mechanical properties in high temperature of ultimate briquettes . Goldsworthy ores (I) and MBR ores were used as iron containing material. The chemical compositions and grain size distribution for the two
5 sampled materials are shown respectively in Table 1 and 2. These constituents were mixed together in a weight parts ratio as shown in Table 3. To the resulting mixture was added a suitable amount of water, and the resulting mixture was homogenized and worked into
10 pellets. The resulting green pellets were cured under the conditions shown in Table 4 for preparation of ultimate pellets. Tables 5 and 6 show the chemical compositions and the results of the tests on the mechanical properties of the resulting ultimate product.
15 As seen from Table 6, the ultimate pellets obtained by the inventive method have practically sufficient properties, notwithstanding the short production time of several days at most.

20

Table 3 mixture ratio of pellets

(in wtoxo)		
	C.B.P. No. 1	C.B.P. No. 2
Ores II (Goldsworthy)	61.0	54.5
Ores (MBR)	26.0	28.5
Polttland cement clinker	9.0	-
Granulated stag of blast frnace	-	7.0
Slaked lime	-	9.0
Calcium carbonate	4.0	-
Light burned dolomite	-	1.0

Table 4 Curing condition for pellets

	C.B.P. No. 1	C.B.P. No 2
Setting	50°C x 2 days	30°C x 3 days
Steam treating	100°C x 1 hr.	100°C x 3 hrs.
Heating	300°C x 1 hr.	250°C x 1 hr.

Table 5 Chemical analysis for pellets

(in wt. %)

	T.Fe	FeO	SiO ₂	Al ₂ O ₃	CaO	HgO	T.S	Ig-loss
C.B.P. No. 1	53.06	0.57	6.71	1.78	8.68	0.63	0.039	4.87
C.B.P. No. 2	52.22	0.29	6.66	1.99	9.65	1.25	0.073	4.62

Table 6 Results of tests on mechanical properties of pellets

	True specific gravity	Apparent specific gravity	Porosity (%)	Compressive strength (kg/p)		JIS reduction test		
				Immediately after pelletizing	After curing	Final reduction percentage (%)	Compressive strength (kg/p)	Swelling index (vol. %)
C.B.P. No. 1	4.4	3.1	28.3	2.0	174.5	88.8	23.9	3.1
C.B.P. No. 2	4.2	2.9	30.6	1.8	151.7	93.7	29.5	0.0

According to the invention, as described above, the ultimate product can be produced in about 3 days from the starting mixture. The ultimate product has sufficient compressive strength to be usable as starting material for iron making. The amount of the binders and other additives can be reduced to generally less than 10 weight percents based on the weight of the material, thus there being no fear of increasing the amount of slags during smelting. Moreover, the inventive method can be practiced continuously by using a simplified device without the necessity of using high pressure steam, thus promoting operating efficiency and reducing the cost of equipment.

Claims:

1. A method of producing cold agglomerates for use in iron making including the steps of preparing a homogenized mixture of powdered or granular iron-containing material, a hydraulic bonding agent and optionally other additives, adding water to said mixture, homogenizing the resulting mixture, mechanically forming the resulting homogenized mixture into agglomerates, and curing the resulting green agglomerates, characterized in that said hydraulic bonding agent is added in an amount within the range from 3 to 10 wt. parts per 100 wt. parts of said iron-containing material, and in that the curing is performed by the consecutive steps of allowing the green agglomerates to stand at ambient temperature during not more than 3 days, steam treating the resulting agglomerates at about 100°C and then air heating the steam treated agglomerates at a temperature from 100 to 500°C.

2. A method of producing cold agglomerates for use in iron making including the steps of preparing a homogenized mixture of powdered or granular iron-containing material, a hydraulic bonding agent and optionally other additives, adding water to said mixture, homogenizing the resulting mixture, mechanically forming the resulting homogenized mixture into agglomerates, and curing the resulting green agglomerates, characterized in that said hydraulic bonding agent is added in amount within the range from 3 to 10 wt. parts per 100 wt. parts of said iron-containing material, and in that the curing is performed by the consecutive steps of allowing the green agglomerates to stand at ambient temperature up to 60°C and in an atmosphere of from 80 to 100% relative humidity during not more than 3 days, steam treating the resulting agglomerates at a temperature from 90 to 100°C and then air heating the steam treated agglomerates at a temperature from 100 to 500°C.

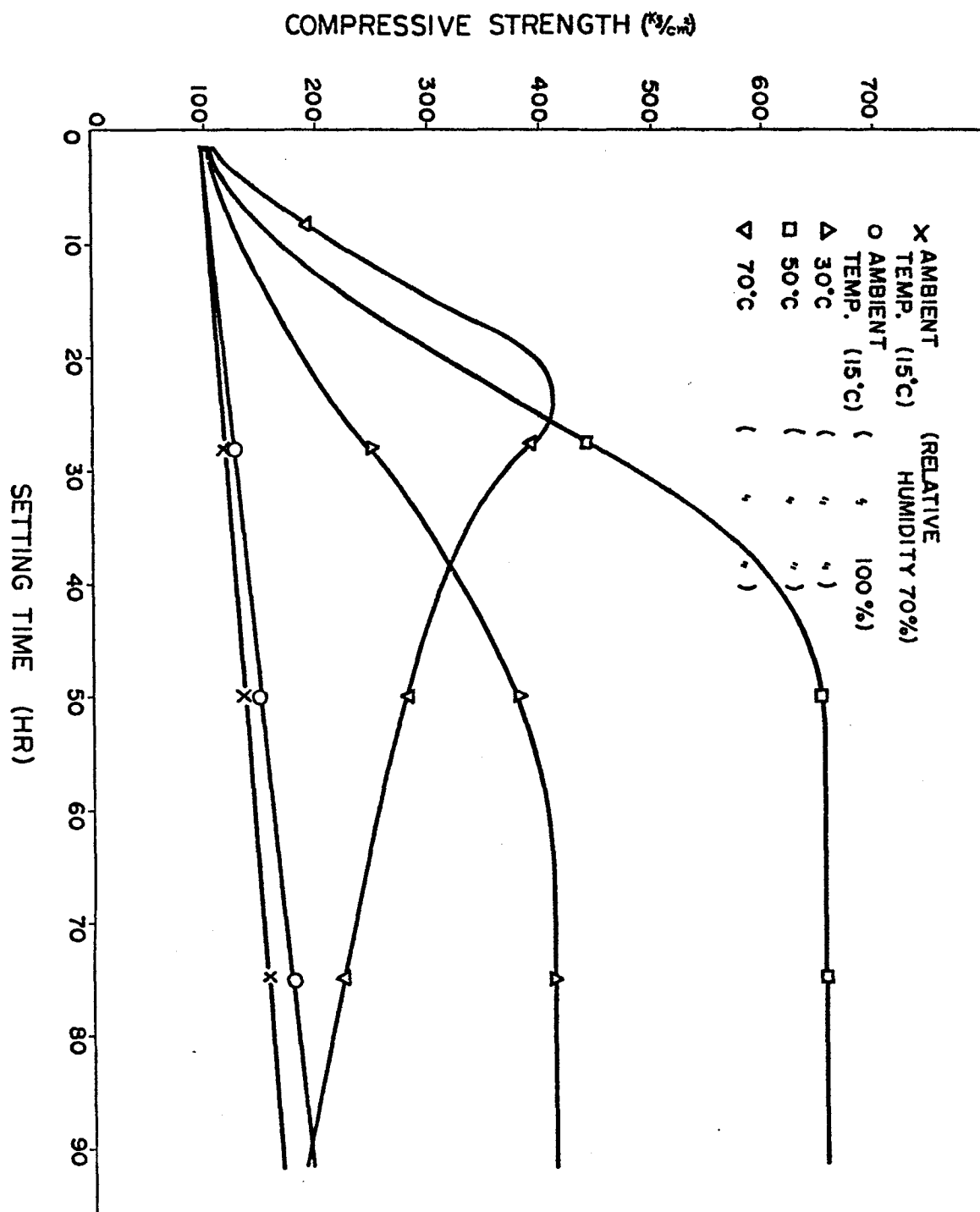


FIG. 1

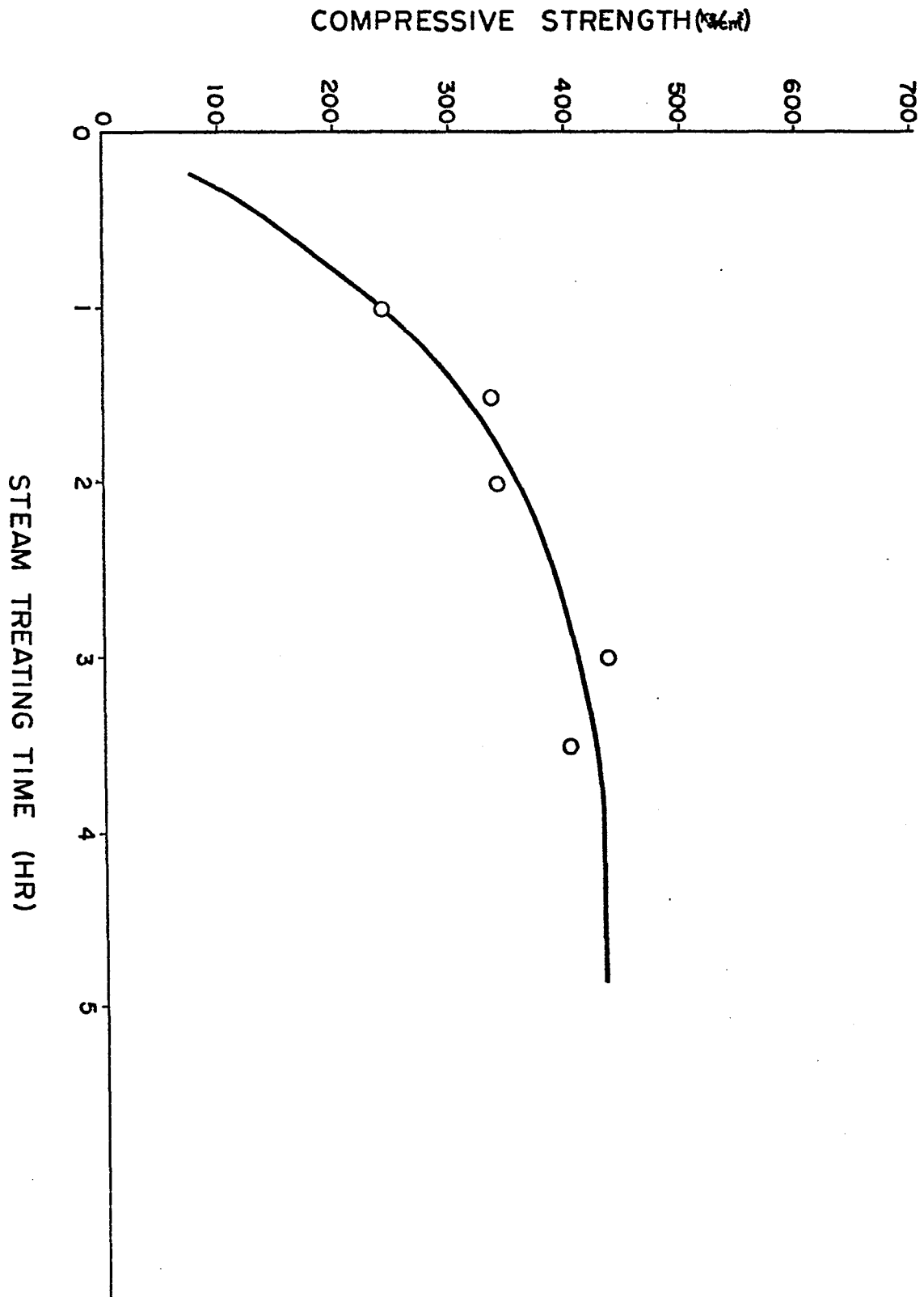


FIG. 2

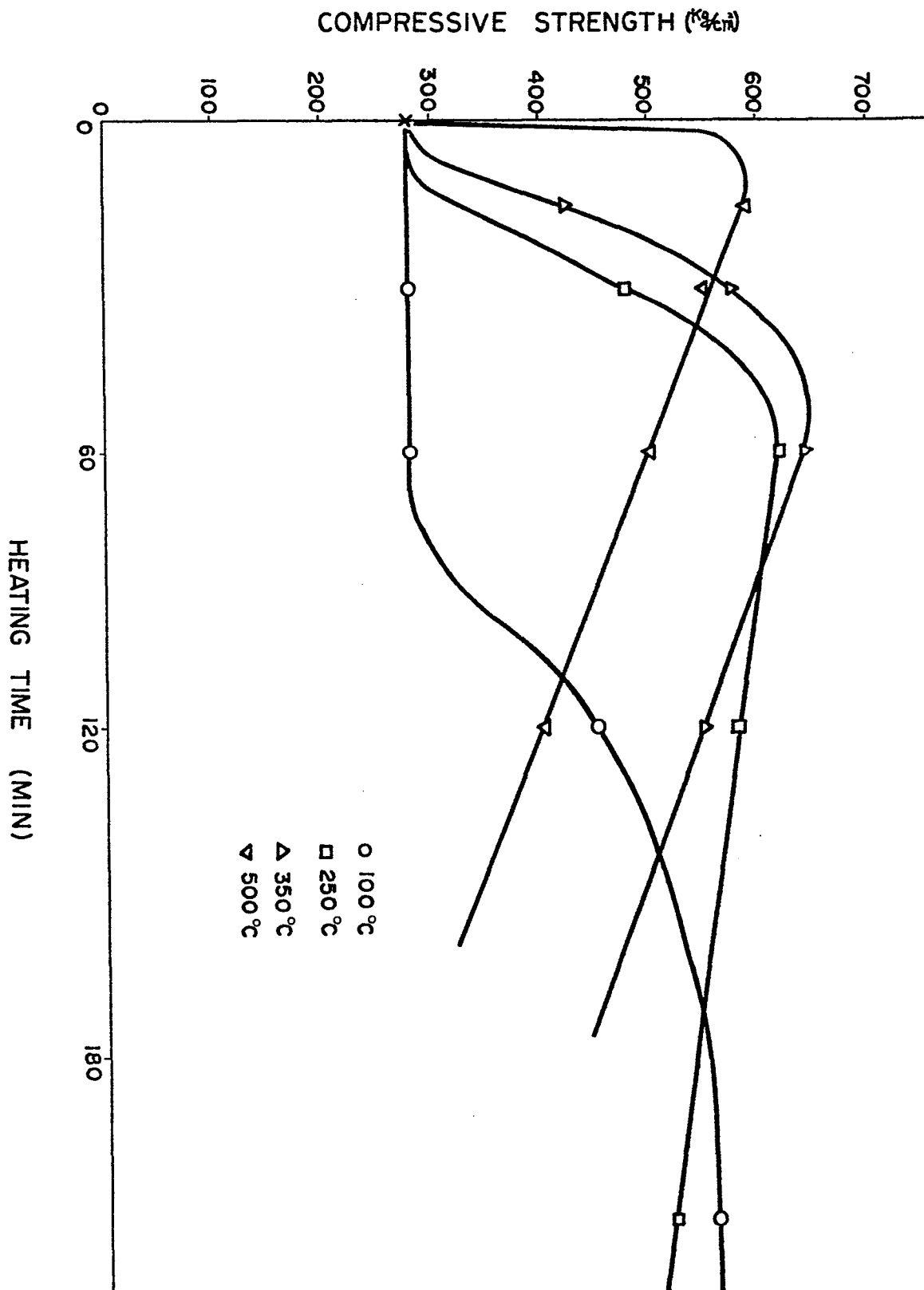
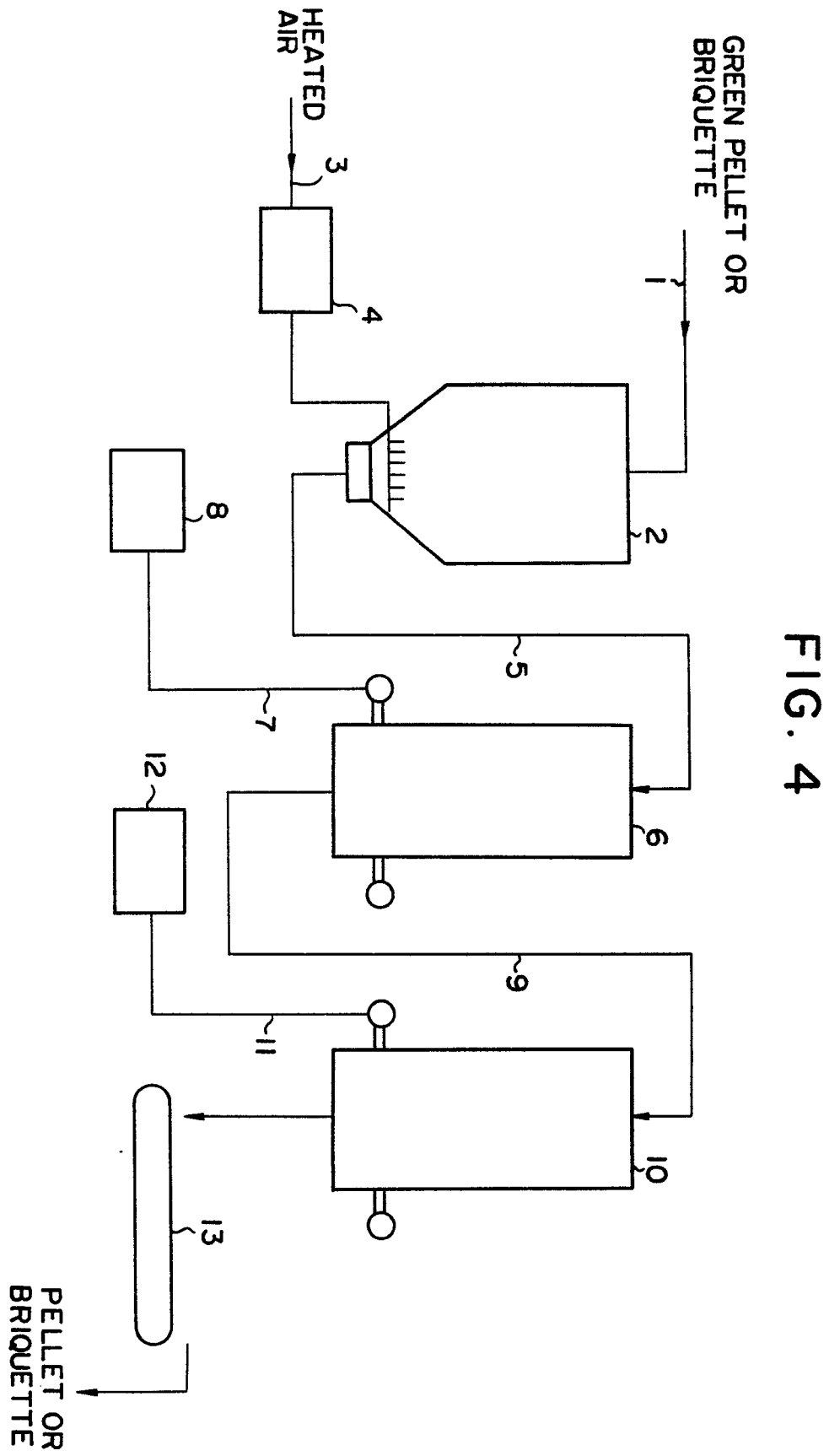


FIG. 3





DOCUMENTS CONSIDERED TO BE RELEVANT			CLASSIFICATION OF THE APPLICATION (Int. Cl. ²)
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	
AD	<u>US - A - 4 049 435</u> (V.E. LOTOSH)		C 22 B 1/243
A	<u>US - A - 3 895 088</u> (M.A. GOKSEL)		
A	<u>FR - A - 2 308 688</u> (BOARD OF CONTROL OF MICHIGAN TECHNOLOGICAL UNIVERSITY)		
A	<u>FR - A - 2 192 172</u> (GUTEHOFFNUNGS-HUETTE STERKRADE A.G.)		
A	<u>FR - A - 2 111 711</u> (A.C. COLD-BOUND PELLETS)		
A	<u>FR - A - 1 085 701</u> (METALLGESELLSCHAFT A.G.)		TECHNICAL FIELDS SEARCHED (Int.Cl. ²)
A	<u>GB - A - 990 672</u> (KENNEDY VAN SAUN)		C 22 B 1
----			CATEGORY OF CITED DOCUMENTS
			X: particularly relevant A: technological background O: non-written disclosure P: intermediate document T: theory or principle underlying the invention E: conflicting application D: document cited in the application L: citation for other reasons
			&: member of the same patent family, corresponding document
The present search report has been drawn up for all claims			
Place of search	Date of completion of the search	Examiner	
The Hague	11-05-1979	JACOBS	