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(54) Title: AGGLOMERATION OF IRON OXIDE WASTE MATERIALS

(57) Abstract

An agglomerate for use in the conversion of iron oxide waste materials to valuable metallic products, the agglomerate at least comprising an iron oxide waste material, a source of free calcium ions, a binder, and water, wherein the iron oxide waste material contains an amount of iron hydroxide. The amount of iron hydroxide is preferably at least 10 % of the iron oxide waste material, and the iron oxide waste material is preferably the iron oxide produced as a by-product in the modified Becher process for the production of synthetic rutile.

AGGLOMERATION OF IRON OXIDE MATERIALS

Field Of The Invention

The present invention relates to the agglomeration of iron oxide materials. The invention also relates to the agglomerates per se produced during the
5 agglomeration of the iron oxide materials, and the use of the agglomerates to produce valuable metallic products.

Background Of The Invention

Several metallurgical processes in many areas of the mining industry generate iron-rich, solid by-products. Reclamation of these by-products is economically
10 desirable because of the valuable iron contained therein and also due to the concomitant elimination of the problem of the disposal of such by-products.

It has previously been found desirable, and in some cases essential, to agglomerate the often finely divided by-product particles into relatively large particles so that they can be conveniently handled and transported, especially
15 when they are to be recycled as a charge to a steel making process.

Various processes have been proposed for agglomerating these by-products. However, most of these processes have met with only a limited degree of success, usually due to the poor structural characteristics of the resultant agglomerates. In particular, a series of work was conducted in the 1960's and
20 1970's by, or in conjunction with, the Michigan Technological University, which culminated in the grant of several patents such as US 3,235,371, US 3,770,416, and US 3,895,088.

The present invention has two main aims. The first is the production of an agglomerate which exhibits particular strengths under particular conditions, and the second is to be able to utilise iron oxide waste materials that have
25



traditionally been considered to be unusable to form such agglomerates. With this in mind, it will be appreciated that the most preferred use of the agglomerates of the invention is in the conversion of the iron oxide waste materials in the agglomerates to valuable metallic products.

5 Summary Of The Invention

The present invention provides an agglomerate for use in the conversion of iron oxide waste materials to valuable metallic products, the agglomerate at least comprising an iron oxide waste material, a source of free calcium ions, an alumino-silicate or alumino-silicate containing binder, and water, wherein the iron
10 oxide material contains at least 10% iron hydroxide, is substantially evenly sized and has a particle size of less than 100 micron.

Description Of The Invention

Iron oxide waste materials predominantly contain iron oxides such as haematite, magnetite, goethite, lepidocrocite, and maghemite, either singly or in
15 combination. Of these mineral species, some are oxyhydroxides such as goethite (α -FeO.OH) and lepidocrocite (γ -FeO.OH). In the present invention, an amount of these oxyhydroxides must be present, and an amount of at least 10% of the iron oxide material is in the iron hydroxide form. In this respect, it is believed that the more common oxides (haematite and magnetite) normally
20 present in iron oxide waste materials do not participate chemically in the bonding reactions.

While the amount of iron hydroxide required is dependent on the other components of the agglomerate, an amount of at least 30% of the iron oxide waste materials is generally more preferred. Indeed, it is possible for all of the
25 iron oxide waste materials to be present in the iron hydroxide form.



The iron oxide material may be the iron oxide derived from the chemical conversion of ilmenite to produce synthetic rutile or upgraded ilmenite, and in one particular form is the iron oxide produced as a by-product in the modified Becher process for the production of synthetic rutile. The iron oxide material in
5 this particular form is a precipitation product which contains amounts of ammonium chloride, the ammonium chloride believed to be a factor in this material normally being disregarded for use in processes such as those of this invention. Such processes (the chemical conversion of ilmenite) result in waste materials that include lepidocrocite and magnetite, and thus they have been
10 found to be particularly useful with the present invention.

However, it will be appreciated that the process of the invention is not to be limited only to the use of this material as a raw product. For example, other sources of iron oxide materials may similarly be used, such as other synthetic rutile processes which use hydrochloric acid (or other leachates), the pickling
15 baths used by the steel galvanising industry, or any other similar sources, preferably where the iron oxide is also a precipitation product.

Another source for the iron oxide material may be the so-called red mud that is an undesirable waste product of the Bayer process for the production of alumina. During the digestion phase in the Bayer process, the alumina in
20 bauxite is dissolved and the remaining material (which is predominantly sand and iron oxide) settles as sludge (or 'red mud'). This red mud is later removed, treated and usually discarded, and generally becomes a waste product that is difficult to easily dispose of. It is envisaged that the red mud produced by the Bayer refineries in Western Australia, Australia is particularly conducive for use
25 in the agglomerates of this invention, particularly as it contains goethite which provides the necessary iron hydroxide.

The free calcium ions in the agglomerates of the present invention are required in order to participate in the formation of complex calcium aluminates, calcium silicates, iron alumina silicates, and hydrates thereof which assist in providing



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high strength bonds. Typically, the free calcium ions would be derived from hydrated lime, but other sources such as calcium oxalate, and calcium chloride could also be used. In particular, where lime is the source of free calcium ions, an amount of lime must be present in a hydrated form, or possibly a readily hydratable form, either as quicklime (CaO) or the hydrate form (Ca(OH)_2) of lime. In this respect, the lime only needs to be in the hydrated form at the point of mixing of the components of the agglomerates, and thus it may be possible to use a readily hydratable lime that is capable of hydrating to a sufficient level on contact with water and on commencement of mixing. In this respect, the particle size of a hydratable lime product may influence its selection and usability herein, as a fine particle size will tend to more rapidly hydrate.

It will also be appreciated that such a hydrated lime product may normally contain amounts of magnesium oxides and hydroxides, and those amounts may range from being insubstantial to being substantial. However, it is envisaged that the amounts of magnesium oxides and/or hydroxides present will not affect the functioning and purpose of the hydrated lime, and may in fact assist, or play the same or similar role.

The iron oxide material in the agglomerate of the present invention is preferably fine and substantially evenly sized. While acceptable agglomerates will be obtained using coarser materials, the effectiveness of the binding mechanism will be reduced, and more of the binding agent will be required. Indeed, the material is preferably characterised by a very small particle size, typically less than 100 micron, although preferably less than 10 micron, with an average particle size of 1 to 2 microns, which results in the binding mechanism being particularly effective.

The agglomerate preferably contains a binder to assist in the provision of mechanical strength during the handling and heating thereof. The binder is preferably an alumino-silicate or an alumino-silicate containing material such as a clay mineral, or a silicate such as sodium silicate, or an aluminite such as



calcium aluminate, or any combination of these, but may also include organic binders such as starches, pitches, resins and the like, although clay minerals such as bentonite, saponite, attapulgite and kaolinite have been found to be particularly useful, on their own or in combinations thereof, with kaolinite being
5 the binder of preferred choice. Additionally, it has been found that the red mud referred to above is able to act as a suitable binder, either alone or in combination with any of the abovementioned binders. The red mud may be used in this manner in conjunction with its use as a source of iron oxide, or it may be the only use of red mud in the agglomerates. In this respect, the red
10 mud contains clay-type minerals and goethite (an iron hydroxide), and so its use may satisfy various of the requirements of the invention.

The agglomerates of the invention preferably contain a water content that is somewhat higher than would traditionally be considered for this type of agglomerate. In this respect, the water content is referred to as the weight of
15 water as a percentage of the total weight of the agglomerate, and is preferably in the range of 15 to 30%, although is more preferably in the range of 21 to 24%.

The amount of water used is very much dependent on the type of carbon source utilised with the agglomerates, which will be described below. In this respect, lower amounts will be required for carbon sources that tend to be less
20 absorbent, such as graphite, high rank coal and coke breeze, while higher amounts will be required for carbon sources that tend to be more absorbent, such as coal char, low rank coals and the like. This is due to the pores of the particles having to be filled with water before the water will remain on the surface thereof. For example, the preferred range of 21 to 24% referred to
25 above has been found to be useful when a carbon source such as Collie coal is used.

In a particularly preferred form of the invention, the agglomerate also contains a pH-increasing modifier. It is believed that at a pH of greater than about 9.5, and more preferably greater than about 12 (which is achievable in one form of the



invention by the addition of the hydrated lime, or by the separate addition of, for example, caustic soda), the binder and the iron oxide minerals present solubilise to form a surface layer, typically several nanometres thick, of complex calcium and iron alumino-silicates around particles or clumps of particles to strongly bind
 5 the agglomerate, thus providing enhanced properties for the agglomerate. In this respect, the use of the red mud as the source of the iron oxide material is again advantageous, as the alkaline nature of red mud may additionally act as a pH modifier.

In a further form of the invention, the agglomerate may also contain a fibrous
 10 material in order to assist in providing the agglomerate with the required properties. In this respect, it has been found that improvements in the handability of the freshly agglomerated mixture can be achieved by the addition of only small amounts of fibrous material such as waste paper and cardboard pulp, wood fibre from wood chips or wood pulp, and possibly even rags or cloth.
 15 The fibrous material may be added in amounts up to about 2% w/w (dry basis) of the mass of the agglomerate. Preferably, amounts in the order of 0.4 to 0.7 % w/w (dry basis) are used as it has been found that amounts greater than about 1.0 % w/w (dry basis) may produce agglomerates of comparatively low cold strength and also comparatively low hot strength. Also, the fibrous material
 20 preferably contains fibres of short length, although the presence of long fibres would not be critical to the performance of the agglomerate.

In relation to the addition of the fibrous material in the manufacture of the agglomerates, the fibrous material is preferably pulped with warm or hot water to produce a fibre having a moisture content of greater than 80 %, and a preferred
 25 solids content in the range of from 25 g/L to 100 g/L, prior to its addition to the remaining ingredients.

While the presence of the fibrous material provides apparent advantages, it is to be understood that the fibrous material is not essential to the present invention and the present invention is not to be limited thereto.



A variety of carbon sources may also be added to the agglomerate to act as reductants and/or carburisers depending upon the required final use of the agglomerate. Typically, where the agglomerate is intended for use in the production of iron, a carbonaceous reductant may be added. The carbonaceous reductant may be any suitable carbon source, such as synthetic rutile kiln waste char, wood char, coal char, coke breeze, coal, lignite, graphite, petroleum coke, fine carbonaceous dusts, oils, greases, vegetable pulps and wastes, and other uncharred waste or low value products with high carbon content. The amount of carbonaceous reductant used is preferably determined so that the agglomerate will self-reduce when heated to temperatures of about 1000°C, the reasons for which will become apparent below. Of course, the degree of self-reduction will depend on the heating process and the quality requirements of the metallic product to be produced by the use of the agglomerates.

The agglomerate may also contain fluxes that will melt during the subsequent use of the agglomerate (again depending upon the required final use of the agglomerate), to produce a slag which is designed to separate from the metal and enhance the chemical properties of the metal by removing undesirable chemical components. The fluxes may include singly or in combination, limestone, lime, silica, dolomite, fluorspar, spodumene or other lithium bearing minerals, and soda ash or other sodium containing materials. In this respect, the presence of the hydratable lime from above may be included in the flux calculations.

In relation to preferred compositions for the agglomerates of the present invention, the primary relationship of concern is between the iron oxide material and the binder. In this respect, it is possible to provide these two components in a range of ratios of from 20:1 to 3:1 iron oxide material to binder. In particular, higher levels of iron oxide material are obviously more preferable than lower levels, although anything within that range will result in acceptable agglomerates.



Furthermore, the amount of free calcium ions utilised in the agglomerates may similarly be varied through a wide range, and may again be provided as a ratio of binder to free calcium ions of about 20:1 to 100:1. At these amounts, suitable amounts of free calcium ions will be provided to give rise to acceptable strengths. However, where lime is used as the source for free calcium ions, it may also be provided in the agglomerate to function as a flux. In this respect, it would not then be unusual to provide lime in a ratio of about 1:1.5 (binder to lime). As can be seen, in this scenario, a large portion of the lime will be acting as a flux.

10 With regard to the remaining components which may be added to the agglomerates of the present invention, those volumes will generally be dictated by the end use of the agglomerates, and thus the appropriate amounts will be readily determinable by a person skilled in the art.

The agglomerates of the present invention are preferably formed by mixing the abovementioned components in an intimate fashion either as a mixture of slurries and dry solids, followed by dewatering to the desired moisture content for agglomeration, or by mixing the dewatered materials and dry solids and then mixing them with water prior to agglomeration. However, it is preferred to avoid certain types of rotation such as rotating drum type actions when mixing in order to avoid the formation of small pellets in the mixture. Thus, high intensity mixing is preferred in order to provide a mixture which is of relatively uniform consistency, much like a damp homogenous soil.

The mixture may then be agglomerated in a number of ways, such as by the compression of the material between two pocketed rolls to produce a pillow-shaped or other shaped briquette, by pelletising where the material is rolled (typically in a rotating disk) to produce small balls, by tableting using a tableting press where the material is forced into a mould and released from the mould, or by extrusion such that the material is extruded under pressure to produce



deep within the agglomerated particle, and also such that the rate of solid state reduction is itself maximised due to the thorough intimate mixing of the components of the agglomerates. In particular, it is envisaged that the iron oxide and the reductant associate intimately due to the fine nature of the iron oxide. This apparently gives a very high surface area which will increase the rate of the solid state reactions which are generally diffusion controlled. Indeed, a high surface area gives multiple reaction sites and there is not the "shrinking core" problem which usually dominates solid state reactions.

In the subsequent treatment of the agglomerates, it is envisaged that the use of agglomerates in the form of briquettes as a feed source will be preferred as they are simpler to transport, require less sophisticated equipment to introduce them into (for instance) a furnace, and their density and size causes them to fall more readily through the slag on the surface of a metal within the furnace into the metal phase. A briquette also is a simpler medium for handling through pre-heating vessels (such as rotary kilns, packed beds or other devices) than a powder and than smaller, less uniformly-sized agglomerates such as pellets. Further, briquettes will pack in a bed such that there is a good permeability for the passage of gas through the bed, as for example in a shaft furnace or cupola.

Detailed Description Of The Invention

The present invention will now be briefly described in relation to the four examples provided herebelow. However, it must be appreciated that the following description is of preferred embodiments only and is not to limit the generality of the invention as described above.

Example 1

A typical composition of an agglomerate in accordance with the invention is shown in the table below:



	Component	% w/w (dry basis)
5	iron oxide	62.0
	kaolinite	8.0
	calcium hydroxide	13.5
	coke breeze	4.5
	coal char	4.5
	Collie coal	7.0
	cardboard fibre	0.5

10 The iron oxide is that obtained as a by-product of the mineral sands industry, having a typical moisture content in the range of 7 to 16%. It is used without further processing.

15 Kaolinite is a clay mineral obtained as a by-product (overburden) of mining operations in the South-West of Western Australia, Australia, and it is substantially free of sand (silica) containing from 0.1 to 2.0% iron oxide as an impurity. It is dried to a moisture content of less than 3% and milled to a particle size of 100% passing 200 micron.

The calcium hydroxide is obtained by the controlled hydration of calcium oxide, is of particle size of 100% passing 45 micron and is less than 1% moisture.

20 The coke breeze, coal char, and Collie coal are milled such that the particle size is 100% passing 4 mm, and typical particle size distributions for the Collie coal and coal char are detailed below. Further, typical values of moisture content for Collie coal are 18 to 28%, for coal char are 3 to 17%, and for coke breeze are 5 to 15%.

25	Sieve Size	Weight Percent Retained			
		Collie Coal		Coal char	
		Example 1	Example 2	Example 1	Example 2
	-4mm +2mm	56	21	42	17
	-2mm +1mm	18	33	40	46
30	-1mm +0.5mm	11	18	9	14
	-0.5mm +212µm	9	13	5	9
	-212 µm	6	15	4	14



The cardboard fibre is the short fibre fraction derived from recycled cardboard, and its moisture content is typically from 75 to 90%.

The iron oxide dust, the kaolinite, and the calcium hydroxide are combined in a ribbon blender. A slurry of cardboard in hot water (approximately 50 parts water
 5 to 1 part cardboard) is added. Mixing is continued for five minutes and then the carbonaceous materials are added. The moisture content is adjusted to be between 21 and 24%.

The resulting mixture is briquetted on a gravity-fed roll press and the briquettes so formed either allowed to cure at ambient temperature without drying or dried
 10 in a conventional briquette drier.

Example 2

The iron oxide, calcium hydroxide, and carbonaceous material components in this example are the same as described in Example 1, although the compositions are as described in the table below.

15	Component	% w/w (dry basis)
	iron oxide	34.0
	red mud	34.0
	calcium hydroxide	15.5
20	coal char	3.0
	Collie coal	13.0
	cardboard fibre	0.5

The red mud is a by-product of the alumina industry, air dried to a moisture content less than 24%.

25 The iron oxide, red mud, calcium hydroxide and carbonaceous materials are combined in a ribbon blender and a slurry of cardboard in hot water added to obtain a moisture content of between 21 and 25%. Mixing is continued for



approximately fifteen minutes and the material briquetted as described in Example 1.

Example 3

Lime kiln dust is a by-product of the lime industry and consists of a mixture of
5 calcium oxide and calcium carbonate with a particle size of 100% passing 45
micron. In this example, the remaining raw materials, namely the iron oxide, the
kaolinite, and the cardboard fibre specified in the table below, are as per
Example 1.

	Component	% w/w (dry basis)
10	iron oxide	70.0
	kaolinite	12.5
	lime kiln dust	17.0
	cardboard fibre	0.5

15 The raw materials are combined in a ribbon blender and water is added to a
moisture content of 18 to 24%. Mixing is continued until hydration of the lime
kiln dust is complete, and the mixture is briquetted as described in Figure 1.

The briquettes produced in each of Examples 1 to 3 all exhibited acceptable
degrees of green strength, cold strength, hot strength, and hot shock strength,
20 and thus are suitable for use in a method of the type briefly outlined in Example
4 below.

Example 4

This example relates to the use of an agglomerate such as those described
above in examples 1 - 3.

25 (a) A cupola is first operated so as to establish a coke bed at the appropriate
height in the shaft. A quantity of scrap metal is then added along with



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10% by weight of coke which is melted mainly to introduce some heat in the hearth area.

- 5 (b) A briquette produced by any one of Examples 1 to 3 is then charged into the cupola along with 12 kg of coke per 100 kg of briquettes. The reductant is made up of a mixture of synthetic rutile plant recycle char and carbon from other sources.
- (c) As the mixture is heated by the rising gases, the reductant reacts with iron oxide to convert the oxide to the metallic state and produce carbon monoxide.
- 10 (d) Additional heat is produced by adding some of the combustion air at higher levels in the cupola to partly combust some of the rising carbon monoxide gas thus increasing the amount of heat transferred in to the burden.
- 15 (e) The metal and slag formed in the shaft are continuously tapped off from the furnace and cast into pigs.

Finally, it will be appreciated that other modifications and variations may be made that are also within the scope of the present invention.



CLAIMS

1. An agglomerate for use in the conversion of iron oxide waste materials to valuable metallic products, the agglomerate at least comprising an iron oxide waste material, a source of free calcium ions, an alumino-silicate or alumino-silicate containing binder, and water, wherein the iron oxide waste material contains at least 10% iron hydroxide, is substantially evenly sized and has a particle size of less than 100 micron.
2. An agglomerate according to claim 1, including a pH modifier capable of maintaining the pH of the agglomerate above 9.5.
3. An agglomerate according to claim 1, including a pH modifier capable of maintaining the pH of the agglomerate above 12.
4. An agglomerate according to any one of claims 1 to 3 wherein the amount of iron hydroxide is at least 30% of the iron oxide waste material.
5. An agglomerate according to any one of claims 1 to 4 wherein the iron oxide waste material is iron oxide waste derived from the chemical conversion of ilmenite to produce synthetic rutile or upgraded ilmenite.
6. An agglomerate according to claim 5 wherein the iron oxide waste material is the iron oxide produced as a by-product in the modified Becher process for the production of synthetic rutile.
7. An agglomerate according to any one of claims 1 to 6 wherein the iron oxide waste material is red mud, red mud being the waste material generated during the digestion phase of the Bayer process for the production of alumina.



8. An agglomerate according to any one of claims 1 to 7 wherein the source of free calcium ions is hydrated lime.

9. An agglomerate according to claim 8 wherein the hydrated lime is present either as quicklime (CaO) or its hydrate form (Ca(OH)₂).

5 10. An agglomerate according to any one of the preceding claims wherein the particle size is less than 10 microns with an average particle size of 1 to 2 microns.

11. An agglomerate according to claim 10 wherein the binder is selected from the group comprising bentonite, saponite and kaolinite, or any combination thereof.

12. An agglomerate according to any one of the preceding claims wherein red mud is used as a binder, either alone or in combination with any other binder, red mud being the waste material generated during the digestion phase of the Bayer process for the production of alumina.

15 13. An agglomerate according to any one of the preceding claims wherein the agglomerate contains a pH-increasing modifier, and a clay binder in the form of kaolin.

14. An agglomerate according to claim 14 wherein the pH modifier is red mud, red mud being the waste material generated during the digestion phase of the Bayer process for the production of alumina.

20 15. An agglomerate according to any one of the preceding claims wherein the agglomerate also contains a fibrous material.

16. An agglomerate according to claim 15 wherein the fibrous material is selected from the group consisting of waste paper and cardboard pulp, wood fibre from wood chips or wood pulp, and rags or cloth.



17. An agglomerate according to claim 15 or claim 16 wherein the fibrous material is provided in an amount of 0.4 to 0.7 % w/w (dry basis) of the agglomerate.
18. An agglomerate according to any one of claims 1 to 17 wherein the
5 agglomerate includes a carbon source to act as a reductant and/or a carburiser.
19. An agglomerate according to any one of the preceding claims wherein the carbon source is a carbonaceous reductant selected from the group consisting of synthetic rutile kiln waste char, wood char, coke breeze, coal,
10 fine carbonaceous dusts and wastes, and other uncharred waste or low value products with high carbon content.
20. An agglomerate according to claim 19 wherein the amount of carbonaceous reductant used is determined so that the agglomerate will self-reduce when heated to temperatures of about 1000°C.
21. An agglomerate according to any one of the preceding claims wherein the
15 agglomerate also contains a flux.
22. An agglomerate according to claim 21 wherein the flux is, singly or in combination, limestone, lime, silica, dolomite, fluorspar, spodumene or other lithium bearing minerals, and soda ash or other sodium containing
20 materials.
23. An agglomerate according to any one of the preceding claims wherein the water is provided in an amount of 15 to 30% of the total weight of the agglomerate.
24. An agglomerate according to any one of claims 1 to 22 wherein the water is
25 provided in an amount of 21 to 24% of the total weight of the agglomerate.



25. An agglomerate according to any one of the preceding claims wherein the agglomerate is formed by mixing its components in an intimate fashion as a mixture of slurries and dry solids, followed by dewatering to the desired moisture content for agglomeration.
- 5 26. An agglomerate according to any one of claims 1 to 24 wherein the agglomerate is formed by compression between two pocketed rolls to produce a pillow-shaped or other shaped briquette, by pelletising where the material is rolled to produce small balls, by tableting using a tableting press where the material is forced into a mould and released from the mould, or
10 by extrusion such that the material is extruded under pressure to produce something akin to thick spaghetti which is then cut into lengths.
27. An agglomerate for use in the conversion of iron oxide materials to valuable metallic products substantially as hereinbefore described with reference to any one of Examples 1 to 3.

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