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COMPACTING OF PARTICULATE METALS

John H. Mahar, Scotch Plains, and Ronald V. Trense, Metuchen, N.J., assignors to Esso Research and Engineering Company, a corporation of Delaware

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This invention relates to the art of forming briquettes. In particular, it relates to the method of compacting particulate metals solids particles to form rigid shapes, coherent masses, aggregates, or briquettes, as well as to the products produced pursuant to such method.

In relatively recent years in view of diminishing iron ore reserves and the increasing need for iron and steel, consideration of methods for producing iron and steel has again shifted to direct iron ore reduction. The methods most often contemplated involve reduction of finely divided iron ore solids particles, especially oxidic iron ores, while the latter is suspended as a fluidized bed, or beds, in upwardly flowing gases, or mixtures of such gases, and reduced at temperatures below the sintering or fusion temperature of the specific ore.

Generally, the particulate ore solids are staged in separate reduction zones, or beds, and gradually reduced through the several stages of oxidation as the fluidized ore is passed successively from one bed to the next and countercurrently contacted by reducing gases. The ore is thus generally reduced from, e.g., ferric oxide to magnetic oxide of iron, and thence to ferrous oxide, and finely to substantially metallic iron. Upon withdrawal, the metallic iron product, generally from about 80 percent to about 95 percent metallized, can be compacted between opposing members at high pressure and sufficiently agglomerated to form generally porous masses.

Upon discharge from the reduction process the very finely divided iron particles are at high temperatures and must be cooled considerably, e.g., to temperatures below about 400° C. or 500° C., prior to discharge into the atmosphere. Even after cooling to relatively low temperatures, however, a host of problems are presented.

These and other objects are produced in accordance with the present invention which contemplates the use, as binders, of small amounts of certain petroleum oil fractions added to, incorporated, or otherwise admixed with finely divided metals, especially particulate reduced iron metal such as results from a direct fluidized iron ore reduction process. These mixtures of binder and metal are pressed, e.g. between opposing surfaces or members, to form rigid shapes, masses, aggregates, or briquettes. The briquettes are then treated at elevated temperature in an oxygen atmosphere and, thus cured, provide a structure possessing, inter alia, high resistance to crushing. The petroleum oil fractions employed as binders are those characterized by a penetration index (ASTM D5–61) at 77° F. ranging from about 85 to about 100 and a softening point (ASTM D36–26) ranging from about 110° F. to about 140° F. Preferably, however, the softening point ranges no higher than about 115° F.

More preferably, such pertoleum fractions are further characterized as having a specific gravity (ASTM D–71) ranging from about 1.0 to about 1.1, and yet more preferably to about 1.03. The petroleum fractions should also have a pure oil viscosity (ASTM D–102) at 275° F. ranging from about 140 seconds to about 250 seconds, and a ductility or elasticity (ASTM D–113) at 77° F. ranging from about 100 centimeters to about 150 centimeters. Such petroleum oil fractions, quite surprisingly, can be utilized in the formation of briquettes and then cured to produce briquettes possessing outstanding resistance to crushing.

In mixing the petroleum oil fraction with the particulate metals, and particularly in the pressing step, it is preferred that the temperature of the metals be elevated to some extent. In the formation of the briquette the temperature should range from about 75° F. to about 400° F., and preferably from about 200° F. to about 350° F.

To produce good crush resistance, it is necessary to cure the briquettes after their formation. The curing of the briquettes is effected by heating in an oxygen-containing atmosphere, preferably air, at temperatures ranging from abopt 300° F. to about 600° F., and preferably from 350° F. to about 450° F. Generally, heat treatment in air ranging from about 1 hour to about 24 hours, and preferably from about 2 to about 16 hours is adequate to cure the briquettes. Surprisingly, briquettes so cured possess far greater compressive strength than uncured briquettes, or briquettes formed from, e.g. asphalt, pitch or tar.

The reason for the effectiveness of these particular petroleum oil fractions as a binder for particulate metals, especially iron, is not known. The compounds adhere tightly to the surfaces of the metals solids particles and cause the individual metal particles, especially after curing, to adhere or bond tightly together.

In forming the briquettes, a sufficient quantity of the pertoleum oil fraction is added to the particulate metals, or mixtures thereof, so that upon application of force the total mass can be compacted to form aggregates. Generally, from about 0.05 to about 10 percent of the petroleum oil fraction, based on the weight of the metal, is thoroughly admixed with the particulate metal, e.g. reduced iron powder. From about 0.5 to about 4 percent of the pertoleum oil fraction is mixed with the particulate metal in most circumstances to give best results. To provide greatest crush resistance the petroleum fraction should generally be employed in concentration ranging from about 1 to about 2 percent.

Following the mixing step, the mixture of binder oil and metal is pressed between opposing surfaces with force sufficient to produce an aggregate mass or briquette. Suitably, pressures ranging from about 20,000 pounds per square inch and higher, and preferably from about 50,000 to about 200,000 pounds per square inch, are employed. Briquettes formed in such fashion are found to provide, after curing, e.g. far greater resistance to crushing, i.e. compressive strength, than briquettes formed without the use of such binder. The briquettes also are more crush resistant than briquettes formed with conventional residual or polymeric substances. Such briquettes also possess good resistance to back-oxidation and the internal void spaces are considerably lessened.

The following nonlimiting examples and pertinent demonstrations bring out the more salient features and provide a better understanding of the invention.

EXAMPLE 1

Particulate iron, from about 80 to about 95 percent metallized, is withdrawn from a fluidized iron ore reduction process at 1400° F. and cooled under a blanketing layer of nitrogen to less than 400° F.

The particulate iron is admixed with one weight percent of a petroleum oil fraction characterized by a penetration index of 95 and a softening point of 110° F. The specific gravity is 1.0, the pure oil viscosity is 160 seconds, and the ductility is 125 centimeters. Portions of the mixture are then contacted at 75,300 pounds per square inch in a ram-type hydraulic press to form individual briquettes. Tests show that the briquettes crumble under pressures of about 25,670 pounds per square inch.

Certain of the briquettes are then removed from the press and treated in air at 400° F. for sixteen hours.

Tests on the briquettes after the treatment show that the briquettes can withstand a pressure of 41,350 pounds per square inch before being crushed.

In sharp contrast, however, when briquettes are formed under identical conditions except that several generally conventional binders are utilized, the crushing strengths of the briquettes range from a low of about 17,000 to a high of about 26,000 pounds per square inch. The advantages of the present invention are apparent inasmuch as crushing strength is improved approximately fifty percent over conventional binders.

EXAMPLE 2

When briquettes are formed in accordance with Example 1, except that 2 and 4 percent concentration, respectively, of the binder is employed, the crushing strengths of the briquettes are 37,300 and 36,130 pounds per square inch, respectively, after curing.

The crushing strength of the cured briquettes diminishes to some extent when added in the two to four percent range, as contrasted with the use of smaller amounts of the additive. A unique advantage is that good crushing strengths can be obtained with small amounts of the petroleum oil fractions.

In the following illustrative examples, a ram-type hydraulic press is employed. Partially cooled particulate iron metal is admixed with portions of the petroleum oil binder described in Example 1 and the mixture pressed. In the pressing operation, pressures and temperatures are controlled to give various desired levels of uncured or "green strength."

The particulate iron employed is one resultant from a fluidized iron ore reduction process. The iron is from about 80 to about 95 percent metallized and is produced from oxidic iron ores in a process wherein the ore is reduced gradually from ferric oxide to magnetic oxides of iron, thence to ferrous oxide and finally to metallic iron.

The data below are the results of a series of runs. Column 1 merely lists the example number; column 2 the weight percent (wt. percent) of the binder employed; column 3 the forming pressure in pounds per square inch (p.s.i.); and columns 4 and 5 give the crushing strengths, prior to curing (green strength) and after curing, of the briquettes in pounds per square inch. The curing step is effected by heat treating the uncured briquettes at 400° F. in air for 16 hours.

Example No.	Binder Content, wt. percent	Forming Pressure, p.s.i.	Crushing Strength	
			Uncured, p.s.i.	Cured, p.s.i.
3	1	32,300	5,430	11,820
4	1	53,800	15,600	32,800
5	1	96,800	30,110	51,700
6	2	32,300	5,440	11,750
7	2	53,800	12,320	23,900
8	2	96,800	22,130	43,600
9	4	32,300	4,870	11,080
10	4	53,800	7,280	24,170
11	4	96,800	18,400	39,200

These specific embodiments of the invention which have been described are illustrative, only, and the invention is equally applicable to particulate metals and alloys of metals, and to mixtures of such metals.

When the foregoing examples are repeated using particulate nickel, manganese, chromium, lead-antimony alloy, and titanium, respectively, satisfactory results are again obtained.

The reason for the effectiveness of these petroleum oil fractions as binders for metals, especially iron, is not known. The initial compression forms the individual metal particles into coherent bodies or aggregate masses. It is believed that the compounds undergo chemical change and are partially oxidized, or perhaps a type of cross-linking is involved. Perhaps polymeric substances are formed. In the curing step, it is believed that a type of oxidation is involved. Whatever the reason, however, very strong bonding characteristics are produced.

It is apparent that certain changes and modifications can be made in the process, and in the briquette which is formed, without departing the spirit and scope of the present invention.

A feature of the invention resides in the novel class of petroleum oil fractions which are incorporated with particulate metals to form the briquettes. By the use of such compounds, high quality briquettes can be formed which have very high crushing strengths. Moreover, briquettes can be formed which are highly resistant to back-oxidation, water absorption and retention, and the like.

Particulate metals can be contacted between opposing surfaces to form briquettes or aggregates in any of a variety of ways including the use of presses, extrusion apparatus, rolls and the like. Extrusion techniques are also satisfactory in the formation of briquettes and lesser pressures are generally needed than when using, e.g., a ram-type hydraulic press. The petroleum oil fractions of this invention, in themselves, provide a salubrious effect in the actual formation of briquettes or aggregate shapes so that lesser force is required and there is less wear on the metal parts constituting the compacting or extruding apparatus. Particulate metals can also be pelletized via use of appropriate pelletizing apparatus.

Having described the invention, what is claimed is:

1. A process for producing a briquette comprising admixing particulate metals with a petroleum oil fraction having a penetration index at 77° F. ranging from about 85 to about 100 and a softening point ranging from about 110° F. to about 140° F., in concentration ranging from about 0.05 to about 10 percent, based on the weight of the metal, pressing the mixture between opposing surfaces at pressures ranging from about 20,000 pounds per square inch and higher to form a briquette, and then heat-treating the briquette in an oxygen-containing atmosphere at temperatures ranging from about 300° F. to about 600° F. for a sufficient time to cause a chemical change and bring about a final curing of the said oil fraction which bonds together the particulate metals.

2. The process of claim **1** wherein the petroleum oil fraction is further characterized as having a specific gravity ranging from about 1.0 to about 1.1, a pure oil viscosity at 275° F. ranging from about 140 seconds to about 250 seconds, and a ductility at 77° F. ranging from about 100 centimeters to about 150 centimeters.

3. The process of claim **1** wherein the petroleum oil fraction employed ranges in concentration from about 0.5 to about 4 percent.

4. The process of claim **1** wherein the particulate metal employed in forming a briquette is substantially reduced iron.

5. The process of claim **1** wherein the petroleum oil fraction employed ranges in concentration from about 1 to about 2 percent.

6. The process of claim **1** wherein the briquette is heat-treated for a period of time ranging from about 1 to about 24 hours.

7. The process of claim **1** wherein the briquette is heat-treated for a period of time ranging from about 2 to about 16 hours.

8. The process of claim **1** wherein the pressure employed to form the briquette ranges from about 50,000 to about 200,000 pounds per square inch.

9. The process of claim **4** wherein the particulate metal is from about 80 percent to about 95 percent metallized.

10. The process of claim **9** wherein the particulate metal employed is one resultant from a fluidized iron ore reduction process.

11. A process for producing a briquette comprising admixing particulate metal consisting essentially of from

about 70 to about 95 percent reduced iron with a petroleum oil fraction having a penetration index at 77° F. ranging from about 85 to about 100 and a softening point ranging from about 110° F. to about 140° F., in concentration ranging from about 1 to about 4 percent, based on the weight of the reduced metal, pressing the mixture between opposing members at pressures ranging from about 20,000 pounds per square inch and higher to form a briquette, and then curing the briquette at temperatures ranging from about 350° F. to about 450° F. for a sufficient time to cause a chemical change and bring about

a final curing of the said oil fraction which bonds together the particulate metals.

12. The process of claim **11** wherein the reduced iron is admixed with the petroleum oil fraction and formed at temperatures ranging from about 75° F. to about 400° F., and the resultant briquette is heat-treated at temperatures ranging from about 300° F. to about 600° F.

No references cited.

ROBERT F. WHITE, *Primary Examiner.*

J. R. HALL, *Assistant Examiner.*