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COMPACTED AMMONIUM NITRATE

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This invention relates to a compacted ammonium nitrate material suitable for use as a component in a commercial explosive and to a method for manufacturing such material. It more particularly relates to a compacted particulate ammonium nitrate material that is porous.

Ammonium nitrate is well known as an ingredient in various commercial explosives. In the past various sensitizers such as: nitroglycerine, TNT, DNT, etc., have been used with ammonium nitrate to produce explosives. In recent years non-explosive sensitizers, such as: carbon black, aluminum dust, powdered sulfur, etc., have been widely used. One of the most popular of the non-explosive sensitizers is light grade petroleum distillate, such as fuel oil, because it is readily available, low in cost, and easy to use, particularly in the field. A light grade of petroleum distillate has an additional operational advantage in that it is a much better gas producer than carbon black or similar solid substances.

It has been found that ammonium nitrate particles which are adapted for use in this type of explosive must have sufficient absorbency to retain the sensitizing oil. If the ammonium nitrate particles are not sufficiently porous so that the added oil may be absorbed, the charge cannot be efficiently sensitized and the result is often a shot failure or an ineffective low order detonation.

It is, therefore, an object of this invention to provide a method of treating ammonium nitrate to produce a particulate ammonium nitrate having a high absorptive capacity for fuel oil.

It is a further object of this invention to provide a particulate ammonium nitrate characterized by decreased density and increased oil absorbency.

The foregoing objects and others, which will hereinafter appear, are accomplished by the method of this invention wherein a starting material of particulate, substantially nonporous ammonium nitrate, is pressed together with sufficient force to make the particles adhere to form compacted sheets or blocks. The so-formed compacted products are agglomerates of separate particles of ammonium nitrate held together by cohesion to form unitary self-supporting sheets. The compacted product is then broken into particles of the desired size. The particulate product is preferably screened to remove the fines and obtain a more uniformly sized product. The sized particles may be further treated with a parting agent to prevent them from adhering together. The particulate product may be sensitized with a sensitizing agent such as a light grade of petroleum to form an explosive mixture. Preferably such explosive mixture is comprised of the particulate ammonium nitrate product and 6 to 8% by weight of a light grade of petroleum distillate.

Conveniently the compacting process may be carried out by means of compacting rolls or by means of a compacting or pelleting press. The use of compacting rolls is usually preferred in the practice of this invention because such process is easily adapted to a continuous process for the production of compacted products and close control over the compacting process may be maintained over an entire production run. Usually in operations utilizing the compacting roll process, the particulate ammonium nitrate starting material is pressed between rotary rolls preferably having slightly roughened surfaces

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to enable the rollers to perform a gripping action on the feed material. The product of the rolls is a continuous thin sheet of compacted ammonium nitrate. The thin sheet is brittle and easily ground by any suitable milling process. In operations utilizing a compacting or pelleting press, the ammonium nitrate starting material is pressed into compact cohesive pellets or blocks and then ground.

Although this process is adaptable to ammonium nitrate materials having various particle densities, it is particularly applicable to ammonium nitrate materials having a particle density greater than about 1.60, and it is most useful with ammonium nitrate starting materials which are essentially nonporous. Ammonium nitrate material having a particle density of about 1.60 or less often has a satisfactory oil absorption capacity and may be sensitized without being subjected to the herein described treatment.

The ammonium nitrate starting material is compacted to form a coherent mass that is later ground to form a particulate ammonium nitrate product. It is generally preferred that the pellet or sheet density of the compacted product be between about 1.10 and 1.65. Products so compacted will generally be found to have a bulk density in the range of between about 0.70 and 1.00 after grinding the sizing so that substantially all of the ground product will pass through a 6 mesh (U.S.S.) screen and be retained on a 20 mesh (U.S.S.) screen. The particle density of such product will usually be between about 1.40 and 1.60.

The term "particle density" as used herein refers to the density value determined by the following procedure: The bulb of a tared pycnometer of known volume is filled with a sample of the ammonium nitrate material. The weight of the filled pycnometer is determined. The bulb closed and a vacuum drawn on the bulb and contents for 5 minutes. The vacuum is then cut-off and a supply of mercury allowed to flow into the bulb. The bulb is again evacuated this time for a period of 3 minutes. With occasional shaking of the bulb, the steps of allowing mercury to flow into the bulb and evacuating the bulb are repeated until no further air is removed. The total weight of the pycnometer containing the ammonium nitrate sample and mercury is determined by weighing. The weight of the ammonium nitrate sample is determined by subtracting the weight of the empty pycnometer from the weight of the pycnometer filled with the ammonium nitrate sample. The volume of the ammonium nitrate is determined by first determining the volume of the added mercury by dissolving the ammonium nitrate from the ammonium nitrate-mercury mixture by adding water, separating the mercury and measuring its volume. The volume of the mercury is then subtracted from the volume of the pycnometer to give the volume of the ammonium nitrate sample. The particle density is then calculated by dividing the weight of ammonium nitrate in the bulb in grams by the volume occupied by the ammonium nitrate in milliliters.

The term "bulk density" as used herein refers to the density value determined by the following procedure: A portion of about 100 ml. of the ammonium nitrate sample to be tested is placed in a dry, weighed 300 ml. cylinder having a 2" inside diameter. A flat-faced, cylindrical 16 pound weight, having a cross-sectional area slightly less than the inside diameter of the cylinder is lowered gently on the surface of the ammonium nitrate material contained in the cylinder. The weight is allowed to remain atop the ammonium nitrate material for a period of 30 seconds. The pressing operation is repeated with successive increments of ammonium nitrate material until the pressed material occupies a volume greater than 300 ml. The material in excess of 300 ml. is removed and the cylinder and contents weighed. The bulk density is

then calculated by dividing the weight of the pressed ammonium nitrate material in grams by 300 ml.

The terms "pellet density" or "sheet density" as used herein refer to the density value determined by the following procedure: The pellet or sheet is weighed and the volume determined by measurement. The pellet or sheet density is then calculated by dividing the weight in grams by the volume in milliliters.

Although a suitable product can be obtained using a feed material at normal room temperature, it is found that a good cohesion of the particulate starting material may be obtained when the starting material is warm. Feed material having a temperature range of from ambient to 200° F. produces a suitable compacted product having satisfactory oil absorbency characteristics. A preferred compacted product is produced when the starting material has a temperature in the range of 140° to 190° F.

Preferably the apparatus in which the above described compacting process may be carried out comprises a pair of coating parallel oppositely rotating rolls. The particulate feed material is gravity fed into the nip or trough of the rolls at a rate sufficient to keep the nip filled with feed material. Compression of the material to the extent that a sheet product having a thickness of between 0.09 and 0.14 inch has been found to be preferred for most products. A compacted sheet having a thickness in the above range is found to be an agglomerate of small particles of ammonium nitrate compressed together to form a unitary self-supporting sheet. Products, according to the invention are usually found to be self-supporting at least to the extent that a section 6" x 6" may be held in a horizontal position by one corner and be self-supporting. Above about 0.14 inch in thickness, the mechanical strength of the sheet is weakened and the mass of such sheet prevents self-support. It has been found that when sheets of a thickness greater than about 0.14 inch were utilized in the process of this invention, the later grinding step reduces a large portion of such product to particles having an undesirably fine size. If the sheet thickness is below about 0.09 inch, it has been found that the sheet product fractures longitudinally when leaving the rolls, and by such action, the material tends to clog the rolls with resultant impairment of the compacting operation.

An ammonium nitrate material of the present invention may also be compacted by means of a pelleting or similar type press. Although a thin press product is desired, such as the compacted product from the roll process described above, it has been found that in order to obtain a satisfactory production efficiency in press operations a thicker sheet or block is desirable. The practical limitations in press face size and the batch-type operation include the need for a large expenditure of work in initially loading the starting material into the press, pressing, and subsequently removing the compacted product from the press. The press product, compacted to the desired density is later ground to produce particles of the desired size. It has been found that a satisfactory compacted product may be made by means of a press which produces a pellet or block having a thickness of between about 0.3 and 0.5 inch.

The invention has been described in terms of a compacted sheet or block, produced from a roll or a press operation, but the described invention should not be considered to be limited to such processes. The compacted ammonium nitrate product may be produced by any suitable means that will produce a compacted ammonium nitrate product of the desired density range from a particulate starting material.

The present invention facilitates the use of ammonium nitrate mixtures sensitized with a light grade of petroleum distillate in small diameter (3 inch or less) bore holes. It has been found that in the practice of this invention, ammonium nitrate particles of about the size that substantially all will pass a 6 mesh and be retained on a 20 mesh (U.S.S.) screen are usually to be preferred as a final prod-

uct. Particles of a size less than 20 mesh (U.S.S.) have been found often to have a tendency to cake, and consequently, have poor pouring and loading properties. Particles of a size larger than 6 mesh have generally been found to have less desirable packing characteristics than smaller size particles. Product having a screen analysis such that it largely passes a 6 mesh (U.S.S.) screen and is held on a 20 mesh (U.S.S.) screen has been found to exhibit a desired oil absorbency of about 7% by weight of ammonium nitrate and are of a size that may be poured in a free flowing manner into small diameter blast holes. Successful propagation of ammonium nitrate charges sensitized with a light grade of petroleum distillate in bore holes as small as 1 1/4" in diameter can be obtained using the ground product of this invention. When the ammonium nitrate starting material was ground to the preferred size and field tested, successful propagation could not be obtained in bore holes of less than 3" in diameter.

The ammonium nitrate material in the form of a pressed sheet or block from the compacting step may be ground by any suitable means such as a mill and the ground product sized by any suitable means such as a screening. Ammonium nitrate having a particle size such that it substantially all passes a 6 mesh (U.S.S.) screen and is held on a 20 mesh (U.S.S.) screen is to be preferred as the ground product.

The ground product is preferably treated with 2 to 3% of a parting agent so that under ordinary conditions of transportation and use, the particles will tend to remain in discrete particle form. Suitable parting agents are diatomaceous earth, fuller's earth, infusorial earth and the like.

Although oil is the preferred sensitizing agent for many applications, the products of this invention may be sensitized by other sensitizing agents such as pulverized coal, carbon particles, finely divided metals such as aluminum or magnesium, TNT, nitroglycerin and the like.

The following table, Table I, shows a comparison of the physical properties of a typical ammonium nitrate starting material and a typical compacted and ground product of the described invention. The starting material is an example of the product of the Stengel process for producing ammonium nitrate by a grinding operation on a cast sheet. A typical ground starting material ranges in size from about that which will pass a 10 mesh (U.S.S.) screen to about that which will be retained on a 120 mesh (U.S.S.) screen. The typical finished product shown was compacted by means of rolls and subsequently, ground and sized. This product is also shown as Example 3 in Table II below.

The oil absorbency values shown in both Tables I and II were determined by the following procedure: A 10 gram sample of the ammonium nitrate material to be tested is placed in a dry 50 ml. beaker. Ten ml. of diesel fuel oil is added atop the material and allowed to percolate therethrough for exactly 10 minutes. The oil-soaked ammonium nitrate is then transferred to a porcelain Büchner funnel (6.1 cm. I.D.) equipped with a Whatman #1 filter paper. A suction is applied to the funnel by means of a water aspirator for exactly 3 minutes and 20 seconds. The suction employed is 6" of mercury for the first 10 seconds and 5 1/2" of mercury for the remainder of the time. The ammonium nitrate material is then transferred to a weighed watch glass. The weight of the oiled ammonium nitrate material is calculated from the combined weights of the material and the watch glass. The percent oil absorption is calculated by the following formula:

$$\text{Percent oil absorption} = \frac{(\text{wt. oiled material in gms.}) - (\text{wt. of original sample in grams})}{(\text{wt. of original sample in grams})} \times 100$$

$$= (\text{gain in wt. of AN material in grams}) \times 10$$

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Table I

Screen Analysis (U.S.S.)	Starting Material	Compacted and Ground Product
Percent on 8 Mesh.....	0.0	11.9
Percent on 10 Mesh.....	0.0	20.2
Percent on 14 Mesh.....	0.8	30.9
Percent on 20 Mesh.....	18.9	32.4
Percent on 35 Mesh.....	24.3	-----
Percent on 45 Mesh.....	13.6	-----
Percent on 70 Mesh.....	18.9	-----
Percent on 100 Mesh.....	8.8	-----
Percent on 120 Mesh.....	5.3	-----
Percent in Pan.....	9.4	4.6
Bulk density.....	0.92	0.79
Particle density.....	1.63	1.46
Percent oil absorbency.....	4.6	7.0

The following table, Table II, shows the properties of several examples of ammonium nitrate products of this invention obtained by compacting a starting material such as that shown in Table I. The products of Examples 1 to 2 were obtained by compacting the starting material in a pelleting press. The products of Examples 3-7 were obtained by compacting the starting material between two rotating rolls. The products from the compacting processes were ground and sized to obtain a product having a particle size such that it substantially all passes a 6 mesh (U.S.S.) screen and is retained on a 20 mesh (U.S.S.) screen. The bulk density, the particle density and the oil absorbency of the sized portion was determined using the density and oil absorption determination methods described above. A small percent by weight of a diatomaceous earth parting agent was added to keep the ground product in particle form. A 30 pound portion of the resultant product was field tested using 6-8% by weight diesel fuel oil as a sensitizer. The sensitized material was tested in bore holes measuring between 2½-3 inches in diameter and between 15-17 feet long. Each portion of the material tested was primed with a 2 x 12 stick of 40% gelatin dynamite and the charge detonated with a #6 blasting detonator. For example, in Example 1, the particulate starting material was compacted at a temperature of about 135° F. in a pelleting press. The density of the pellet produced was 1.14. The compacted pellet was then ground and the portion having a particle size such that it substantially all passes a 6 mesh (U.S.S.) screen and is retained on a 20 mesh (U.S.S.) screen separated. The sized portion was found to have a bulk density of 0.75. The particle density was found to be 1.53. The oil absorbency of this product was tested and found to be 7.4%. An explosive mixture was compounded by taking a 30 pound portion of the material, treating it with 2% by weight of diatomaceous earth and sensitizing it with 6% by weight of diesel fuel oil. The sensitized material was loaded into a bore hole, primed and detonated. The shot was good and the average velocity of detonation was 8525 f.p.s. (feet per second).

The other examples shown in Table II were conducted in a similar manner.

Table II

	Example 1	Example 2	Example 3	Example 4	Example 5	Example 6	Example 7
Starting Material: Temperature, ° F.	Ca 135	Ca 155	179	178	129	182	182
Compacted Material:							
Pellet density.....	1.14	1.39	-----	-----	-----	-----	-----
Pellet thickness (inches).....	0.49	0.38	-----	-----	-----	-----	-----
Sheet thickness (inches).....	-----	-----	0.119	0.120	0.110	0.124	0.127
Ground Product:							
Bulk density.....	0.75	0.74	0.85	0.79	0.87	0.81	0.76
Particle density.....	1.53	1.53	1.55	1.56	1.50	1.54	1.56
Percent Oil Absorption.....	7.40	6.37	5.56	6.02	6.44	7.28	7.05
Percent Diatomaceous Earth Added.....	2	2	2	2	2½	2	2
Field Test:							
Percent Diesel Fuel Added.....	6	6	8	8	8	8	8
Feed into bore hole.....	Good	Good	Good	Good	Good	Good	Good
Speed of detonation, f.p.s.....	8,525	7,842	8,399	8,325	7,856	7,083	6,263
Shot Results.....	Good	Good	Good	Good	Good	Good	Good

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By the term "consisting essentially of" as used in the following claims, it is meant to include compositions containing the named ingredients in the proportions stated and any other ingredients which do not deleteriously affect the compositions for the purposes stated in the specification.

What is claimed is:

1. The process of producing a firm, porous, particulate ammonium nitrate material which comprises mechanically compacting a dense, particulate, substantially non-porous ammonium nitrate starting material into a thin, agglomerated, self-supporting sheet, and grinding said sheet to obtain a product comprising low density particles of ammonium nitrate, said particles being suitable for use as a component in ammonium nitrate explosive compositions.
2. The process as described in claim 1 wherein the particles of ammonium nitrate obtained from the grinding step have a light grade petroleum distillate absorptive capacity, of between about 5 and about 8% by weight.
3. The process as described in claim 1 wherein the ammonium nitrate starting material is compacted while the material is between about 140° and about 190° F.
4. The process as described in claim 1 wherein the ground, low density particles of ammonium nitrate are sized to about the size that they will pass a 6 and be retained on a 20 mesh (U.S.S.) screen.
5. The process as described in claim 1 wherein the starting material is compacted by passing the starting material between a pair of rotating rolls.
6. The process as described in claim 1 wherein the starting material is compacted by means of a pair of rotating rolls, the compacted product having a sheet density in the range of from about 1.10 to about 1.65.
7. The process of producing an ammonium nitrate material suited to use in an oil sensitized ammonium nitrate explosive comprising the steps of compacting a particulate substantially nonporous ammonium nitrate starting material ranging from about the size which will pass a 10 and be retained on a 120 mesh (U.S.S.) screen and having a particle density greater than about 1.60 into a compacted sheet, grinding said sheet, and separating the particles that range from about the size that will pass a 6 and be retained on a 20 mesh (U.S.S.) screen, the said separated particles having a particle density range of from 1.40 to 1.60.
8. The process of producing a firm, porous, particulate ammonium nitrate material which comprises mechanically compacting a particulate ammonium nitrate starting material having a density greater than 1.60 into a thin, agglomerated, self-supporting sheet, and grinding said sheet to obtain a product having a light grade petroleum distillate absorptive capacity of between about 5% and about 8% by weight and a particle density of between about 1.40 and about 1.60.
9. An ammonium nitrate explosive composition consisting essentially of the product of claim 8 and a light

grade of petroleum distillate absorbed on the surface of the compacted and ground product.

10. An ammonium nitrate explosive composition consisting essentially of the product of claim 8 and a light grade of petroleum distillate absorbed on the surface of the compacted and ground product, the amount of light grade petroleum distillate absorbed on the ammonium nitrate product being between about 5 and about 8% by weight.

References Cited in the file of this patent

UNITED STATES PATENTS

1,992,217	Kirst et al.	Feb. 26, 1935
2,770,539	Martenet	Nov. 13, 1956
2,773,753	Stengel	Dec. 11, 1956
2,798,801	Kieffer et al.	July 9, 1957
2,904,412	McBride et al.	Sept. 15, 1959