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## EXPLOSIVE

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This invention relates generally to explosives and particularly to blasting explosives of the type containing carbonaceous material as well as to the process of preparing such explosives and the ingredients thereof.

Blasting explosives, such as the low density dynamites of the permissible type generally comprise a liquid explosive, such as nitroglycerine, inorganic explosive salts, such as ammonium nitrate, and a bulky carbonaceous filler. Permissible dynamites are designed particularly for use in mining coal, and their composition is such as to minimize the formation of toxic concentrations of carbon monoxide and to avoid danger of fire and explosion in the mine due to dust.

The density of permissible dynamites can be decreased by increasing the proportion of the bulky carbonaceous ingredient up to about 20%, which constitutes the upper limit since higher percentages cause excessive concentrations of carbon monoxide in the explosion gases. The maximum proportion of filler is further limited, for a given percentage of nitroglycerine in the explosive, to an amount which permits satisfactory sensitivity to detonation, i. e., the explosion of a cartridge must initiate the explosion of a similar cartridge placed some minimum distance away, for example over a gap of three inches between the ends of two cartridges or ten inches between the open ends of two half cartridges. In general, the lower the nitroglycerine absorptivity of a given filler, the less will be the effect of the filler in decreasing the sensitivity of dynamites in which it is used. Loss in sensitivity may be corrected by increasing the percentage of nitroglycerine, but this involves the disadvantages of increased cost of the explosive and higher flame temperature, density, and rate of detonation.

Heretofore various materials have been employed as the carbonaceous filler in such low density dynamites. A great variety of materials of vegetable origin, such as plant stalks, moss, various kinds of wood flour, or sawdust, have been employed. In general, however, such materials are too highly absorbent of nitroglycerine to permit their use in the manufacture of permissible dynamites of very low density as, for example, dynamite of the type such that more than about five hundred 1¼ inch x 8 inch cartridges may be obtained per one hundred pounds of dynamite. While various methods have been heretofore suggested for treating such materials in order to reduce their absorptivity as, for example, by impregnating them with resin, starch or inorganic salts, such treatments frequently increase the

density of the filler and in any event are disadvantageous because of the additional manufacturing cost and difficulty of obtaining a uniformly treated product.

The object of the present invention, generally stated, is to provide a low density dynamite having a carbonaceous filler of plant stalk material whose absorptivity for nitroglycerine is low.

Another object of the present invention is to provide an improved carbonaceous filler for low density dynamite.

A further object of the present invention is to provide a process of treating low density plant stalk material whereby the low density is maintained without rendering the material highly absorptive toward nitroglycerine.

A more specific object of the present invention is to provide a process of preparing carbonaceous filler material of the plant stalk type whereby the cell walls of the stalk are maintained substantially intact and the nitroglycerine absorptivity minimized.

Other objects will become apparent to those skilled in the art when the following description is read.

In accordance with the present invention an explosive composition of the dynamite type containing a carbonaceous filler is prepared with a carbonaceous filler material which, while being substantially void on its interior and thus having a low density, is composed of particles, the surfaces of which are substantially continuous and hence the absorptivity toward nitroglycerine is minimized, such nitroglycerine as attaches itself to the filler being for the most part by adsorption. In accordance with one feature of the present invention the carbonaceous filler material may be prepared from low density plant stalks in such manner that during the process of treatment including subdivision, the cell walls of the stalk are maintained substantially intact and free of rupture or puncture insofar as possible, save along the lines of subdivision. Such preservation of the cell walls structure results in a filler material which, while of low density, is so because of internal voids having no channel of communication with the surface of the particle. Hence, upon the exposure of the filler material to nitroglycerine, the nitroglycerine cannot penetrate to the interior and occupy the voids, but, on the contrary, must confine itself to the surfaces of the particles of filler material.

Such preservation of cell wall structure may be accomplished by subdividing the material by a process of cutting with knives or shearing as dis-

tinguished from the usual process of comminution which involves a grinding, shredding or rasping action. Particularly beneficial results are obtained if the material is cut while containing a substantial percentage of moisture as, for instance, a moisture content sufficient to saturate the cell walls of the material. In the case of most low density annual plant stalks a moisture content of 25% to 50% (on the weight of the oven dry material) is sufficient to saturate the cell walls. The process of preparing filler material in accordance with the present invention is particularly applicable to annual plant stalks of the type having a substantial pith content such, for instance, as the stalks of broom corn, sugar cane, sorghum cane, corn and sunflower.

In accordance with the present invention, after the plant stalk material has been subdivided in the manner, just described, the filler material may be processed by further wetting as, for instance, with water or with steam followed by drying. This treatment results in a slight expansion of the particles as well as a stiffening thereof, the latter result being accomplished apparently by the partial hydrolysis of the hemicellulose compounds of the cell walls. After such hydrolysis of the hemicellulose components and upon drying of the material, the hydrolytic products are left, upon evaporation of the water, at or near the exterior surface of the particles with the result that the cell walls thereafter are impregnated, stiffened and strengthened thereby. The stiffness and strengthening of the filler material is of particular importance in view of the fact that dynamite cartridges are loaded under pressure, and, if the filler material does not possess sufficient stiffness of strength to withstand the pressure of loading, it is apparent that the particles of filler collapse with consequent increase in the density of the explosives.

A convenient device for use in subdividing the plant stalk material in accordance with the present invention in order to effect subdivision without substantially rupturing or puncturing the cell walls except along the lines of subdivision is a device known as a rotary knife cutter. This apparatus consists of a rotor fitted with knives, rotating within a cylindrical casing provided with stationary knives and having perforated sides. The desired particle size is obtained by adjusting the speed of rotation, the clearance between the stationary and rotating knives, and the size of the perforations in the screen enclosing the cutting chamber. The stalks are therefore severed into particles by clean cuts so that the surfaces or edges of the particles are smooth and even and so that the interior walls of the particles remain substantially intact. In operation, particles which have been cut to the desired size are immediately discharged through the screen, particularly if suction is applied around the cutting chamber according to the usual practice, and the desired subdivision is thus obtained with minimum crushing and laceration of the cell walls, and is accompanied by the formation of only a small percentage of fines. In contrast to material which has been comminuted by other means, the rotary knife cutter product is characterized by substantial uniformity in the shape of the particles and by the absence of slivers.

In comparative experiments in the comminution of broom corn stalks for example, the following yields of material passing through an 8 mesh and retained on a 30 mesh screen were obtained:—49% by means of a hammer-mill, the

overs having been re-milled four times; 43% by means of a rasping wheel; and 88% by a single pass through a rotary knife cutter. The percentages of fines were respectively 23%, 25%, and 12% and of overs 28%, 32%, and none for the rotary knife cutter. The product obtained with the rotary knife cutter was free of slivers as contrasted with the other two and flowed more readily. The overs obtained from the hammer milling and rasping treatments consisted of fibrous strands which resisted further subdivision. This fibrous material is retained in the particles of the cutter product, which are considerably strengthened thereby. Similar advantages are gained by the use of the rotary knife cutter over other methods of comminution in the subdivision of other low density plant tissue; for example, screened yields of corn stalk, obtained as above, were 67%, 55% and 85%, respectively. The following table illustrates the differences in characteristics between material subdivided in accordance with the present invention and that subdivided by other means of comminution:

| Material           | Percent H <sub>2</sub> O | Method of subdivision | Yield 8/30 mesh product | Density | Nitro-glycerine absorption |
|--------------------|--------------------------|-----------------------|-------------------------|---------|----------------------------|
| Broom corn stalks. | 7-10                     | Rotary knife cutter.  | 88                      | 0.133   | 2.90                       |
| Do.....            | 7-10                     | Hammer mill.          | 49                      | 0.122   | 2.23                       |
| Do.....            | 7-10                     | Rasping wheel         | 43                      | 0.115   | 4.08                       |
| Corn stalks....    | 10                       | Rotary knife cutter.  | 85                      | 0.123   | 2.57                       |
| Do.....            | 10                       | Hammer mill.          | 67                      | 0.124   | 2.38                       |
| Do.....            | 10                       | Rasping wheel         | 55                      | 0.135   | 2.67                       |

As pointed out hereinbefore, it is advantageous that the plant stalk material be cut in a moistened condition. For instance, in the case of broom corn stalks, optimum values of density and nitroglycerine absorption are obtained when the material is cut at a moisture content greater than 25% of the weight of the oven dry material. 25-50% moisture by weight of oven dry material commends itself as this moisture content is sufficient to reduce the brittleness and hence inhibits tearing and puncturing of the cell walls while it is not so great that substantial amounts of free water will be present, and consequently sticking and clogging of the cutting apparatus is avoided. In the case of broom corn stalks, at a moisture content of about 30%, substantially no free water is present, the moisture being almost entirely absorbed by the cell walls. The following table illustrates the effect upon the resultant product of various moisture contents during the process of subdivision by cutting means with reference to both broom corn stalks and corn stalks:

| Material                | Percent H <sub>2</sub> O | Density | Nitro-glycerine absorption |
|-------------------------|--------------------------|---------|----------------------------|
| Broom corn stalks ..... | 7                        | 0.133   | 2.90                       |
|                         | 18                       | .080    | 2.67                       |
|                         | 33                       | .086    | 1.55                       |
|                         | 75                       | .092    | 1.71                       |
| Corn stalks .....       | 10                       | .123    | 2.57                       |
|                         | 30                       | .127    | 1.67                       |
|                         | 75                       | .097    | 1.62                       |

Material such as broom corn stalks which has been cut wet, as hereinbefore described, may be further improved by an after treatment with about an equal weight of water followed by drying at a temperature from 100-150° C. As

pointed out hereinbefore, this treatment results in expansion of cells with consequent decreased density of the product and further results in a strengthening of the particles, probably on account of the hydrolysis of the hemicellulosic components of the cell walls through the action of heat and moisture. This treatment effects a substantial decrease in density. As illustrating the effect of such after treatment using varying percentages of water or steam, the characteristics of broom corn which had been initially cut at a 33% moisture content are shown in the following table:

| Percent moisture added | Density | Nitro-glycerine absorption |
|------------------------|---------|----------------------------|
| 33.....                | .086    | 1.56                       |
| 100.....               | .072    | 1.75                       |
| 150.....               | .071    | 1.79                       |
| 500.....               | .067    | 1.64                       |

As compared with a known carbonaceous filler material consisting of expanded flaked cereal grain, it may be pointed out that the density of such flaked cereal grain product is 0.091 while the nitroglycerine absorptivity characteristic is 2.83 according to the method used in determining those characteristics as set forth in the foregoing tables.

In the foregoing tables the density values were determined by filling a tared glass cylinder of known volume, 40 mm. in diameter and 250 mm. high, with the material, which was compressed during the filling operation at  $\frac{1}{4}$ ,  $\frac{1}{2}$ ,  $\frac{3}{4}$ , and full measure by means of a closely fitting wooden plunger carrying a 15 pound weight, and weighing.

In the foregoing tables the nitroglycerine absorptivity values were determined by the following method: About 25 gms. of filler are mixed with 75 gms. of nitroglycerine and stored for 2 hours at 100° F. with occasional stirring. A portion of the mixture is then centrifuged in a tared Gooch crucible at 600 R. P. M. for 5 minutes and the crucible and contents weighed. The nitroglycerine is then extracted with ether and the crucible and contents are dried and weighed. The absorption value is then calculated by dividing the weight of nitroglycerine absorbed, by the weight of material (corrected for loss in weight during a blank ether extraction).

As an illustrative example of the present invention the stalk of various varieties of broom corn (Standard, Western Dwarf, or Whisk Dwarf), the brush of which is utilized in the manufacture of brooms, may be employed as the raw material. The stalks may be cured in the field after the harvesting of the broom and are then stored to be used as needed. The stalks are passed through an ensilage cutter and cut to short lengths of from a fraction of an inch to several inches. The stalk segments are thoroughly sprayed with water and allowed to stand in heaps for at least an hour. They are then spread out in a thin layer and allowed to drain and air-dry for about 12 hours, or for a shorter time if a forced air draft is used, until the moisture content is about 25 to 40% of the oven-dry weight of material. A rapid rate of drying is preferable since then the outermost layer of the stalks becomes dried to a lower moisture content than the interior pithy portions, facilitating the cutting action. The segments are then fed into the hopper of a rotary knife cutter in which the

speed of rotation, clearance between knives, and the size of the screen perforations are adjusted so as to yield a product of the desired granulation, with minimum production of fines. The material is then sprayed with an equal weight of water or mixture of water and steam and then dried at temperatures between 100° and 150° C. by means of apparatus, such as a continuous plate or rotary dryer, which serves to keep the material well stirred, loose, and substantially free from any crushing, to a moisture content of about 3%. The product is screened by customary means, a yield of over 80% of particles passing through an 8 mesh but retained on a 30 mesh screen being obtained. The product has sufficiently low density and low absorptivity toward nitroglycerine to permit its use as the carbonaceous ingredient in permissible dynamites providing more than 500  $1\frac{1}{4}$  x 8 inch cartridges per 100 pounds. If a product of still lower density is desired, it may be obtained by removing the heavier particles by means of air or gravity separation after the cutting operation and applying the spraying, drying, and screening treatments as described above to the light fraction. The fines which result at the final screening, amount to less than 15% of the raw material and may be utilized as the carbonaceous ingredients of higher density dynamites or for other uses.

The preparation of the product may be conveniently carried out continuously in the following manner. The stalks are passed through an ensilage cutter and the short lengths are treated with excess water, drained, and partially dried while being conveyed to the hopper of a rotary knife cutter, the resulting particles being discharged into a cyclone collector. The material is then passed through mixing apparatus of the screw conveyor type, being subjected to a fine spray of water or wet steam near the charging end, preferably adjusted to treat the particles with about an equal weight of water. The material is then passed through drying apparatus, which may be a continuous plate dryer of the type in which the material is carried from one end of a heated metal plate to the other by means of intermittently operating scrapers. The time of travel through the apparatus may be adjusted according to the plate temperature, for example one hour for a plate temperature of 130° or longer periods for lower temperatures. After the treatment in the dryer, the particles at a moisture content of about 3 are transferred by means of an air blast, which serves also to cool the material, to a screening apparatus, and the product passing through an 8 mesh and retained on a 30 mesh screen is stored or packed for use in low density explosives. During the steaming and drying treatments, the color of the product may become deepened because of partial caramelization.

Another waste product of the broom industry which forms a highly satisfactory raw material for the preparation of very low density carbonaceous filler consists of the handle or stem of the brush, commonly called "broom chips", which are obtainable from broom factories. This material comprises one to twelve inch lengths taken from between the head, or broom, and the uppermost node of the broom corn stalk and is suitable in its entirety for the preparation of very low density filler by the above described process, since the relatively dense horny layer enclosing the pith is very thin.

The product thus prepared may be employed as a carbonaceous filler ingredient in any well known dynamite formula such, for instance, as those illustrated in the following table, it being understood that the usual methods of incorporation, loading, etc., are followed:

|                                       | A    | B    |
|---------------------------------------|------|------|
| 10 Nitroglycerine.....                | 13.0 | 15.0 |
| Coarse $\text{NH}_4\text{NO}_3$ ..... | 68   | 59.5 |
| Fine $\text{NH}_4\text{NO}_3$ .....   | 5.0  | 5.5  |
| $\text{NaNO}_3$ .....                 | 13.5 | 19.5 |
| Carbonaceous filler.....              | 0.5  | 0.5  |
| Chalk.....                            |      |      |

As illustrating the cartridge count and sensitivity of dynamites prepared according to the foregoing formulae and employing carbonaceous filler material produced in accordance with the present invention as contrasted with carbonaceous filler material prepared by other methods, the following tables are given, the products prepared in accordance with the present invention being indicated by an asterisk:

Formula A

| 30 Carbonaceous filler                        | No. of 1¼" x 8" cartridges per 100 pounds dynamite | Sensitivity at 70° F. |                |                  |                |
|-----------------------------------------------|----------------------------------------------------|-----------------------|----------------|------------------|----------------|
|                                               |                                                    | Half cartridges       |                | Whole cartridges |                |
|                                               |                                                    | Fresh                 | Stores 60 days | Fresh            | Stores 60 days |
| 35 Broom corn stalk, rasped.                  | 386                                                | Inches 11             | -----          | Inches 2½        | -----          |
| Broom corn stalk, process of this invention.* | 445                                                | 16                    | 14             | 6                | 4              |
| Corn stalk, rasped.                           | 346                                                | 13                    | -----          | 5                | -----          |
| Corn stalk, process of this invention.*       | 401                                                | 17                    | 15             | 7                | 5              |
| 40 Flaked cereal grain product.               | 400                                                | 12                    | 9              | 4                | 2½             |

Formula B

| 50 Carbonaceous filler                        | No. of 1¼" x 8" cartridges per 100 pounds dynamite | Sensitivity at 70° F. |                |                  |                |
|-----------------------------------------------|----------------------------------------------------|-----------------------|----------------|------------------|----------------|
|                                               |                                                    | Half cartridges       |                | Whole cartridges |                |
|                                               |                                                    | Fresh                 | Stored 60 days | Fresh            | Stored 60 days |
| 55 Broom corn stalk, rasped.                  | 482                                                | Inches 10             | -----          | Inches 6         | -----          |
| Broom corn stalk, process of this invention.* | 540                                                | 12                    | 11             | 7                | 6              |
| Flaked cereal grain product.                  | 500                                                | 12                    | 10             | 4                | 2              |

A particularly important characteristic of the carbonaceous filler material prepared according to the present invention and the explosives containing the same is the combined low density and resistance to compression and to moisture. The latter is a particularly important characteristic since the tendency for filler material heretofore employed to lose bulk on exposure to moisture with consequent "mushiness" of the dynamite cartridges is not encountered in explosives prepared in accordance with the present invention.

From the foregoing description it is apparent that the present invention accomplishes its objects and that the explosive compositions formulated with carbonaceous filler materials prepared from annual plant stalks, treated in accordance with the present invention, display marked im-

provements in cartridge count, sensitivity and retention of sensitivity on storage over such explosive composition as are formulated with carbonaceous fillers of the prior art. Furthermore, they enable the production of dynamites according to Formulas A and B, having satisfactory sensitivity and cartridge counts over 400 and 500 respectively, a result which cannot be realized with comminuted plant stalk tissue prepared by prior art methods unless they are subjected to a costly and troublesome impregnation treatment with extraneous substances. Due to the uniformity of particle shape and the absence of slivers in the fillers, the mixing and cartridgeing of the dynamite may be effected with unusual facility and uniformity and the resulting cartridges are characterized by their smoothness and uniform appearance. The dynamites prepared with fillers produced by the process of this invention are much more resistant to the effects of moisture than compositions formulated with the flaked cereal grain product, which is soluble in water to the extent of 35% at ordinary temperatures.

While in the foregoing description a complete disclosure of several illustrative embodiments of the present invention has been given, it is not to be understood that the invention is limited to the details of that disclosure. For instance, while reference has particularly been made to dynamites of the permissible type and to the use of treated broom corn stalks as the filler therein, it is obvious that such filler material may be employed in any type of dynamite, and furthermore that other annual plant stalks, such as the stalks of sugar cane, sorghum cane, corn, sunflower, and the like, may be treated according to the process hereinbefore described, slight variations being permissible in order to adapt the process to the material and produce carbonaceous fillers having optimum characteristics. It is realized that such variations, modifications and applications will present themselves to those skilled in the art without departing from the spirit of this invention, and it is to be distinctly understood, therefore, that such modifications and the use of such individual features, combinations and subcombinations of the features of the present invention as do not depart from the spirit of this invention are, although not specifically described herein, contemplated by and within the scope of the appended claims.

Having thus described the invention, what is claimed is:

1. A low density carbonaceous ingredient of an explosive composition, consisting of clean-cut particles of annual plant stalk, said particles having a density, when compressed at 12 pounds per square inch, between 0.075 and 0.150 and sorbing when saturated not more than 2.00 times its weight of nitroglycerine.

2. An explosive composition comprising a liquid sensitizing explosive, explosive salts, and a low density carbonaceous ingredient consisting of particles of clean-cut broom corn stalk which has a density when compressed at 12 pounds per square inch between 0.075 and 0.150 and sorbs at saturation not more than 2.00 times its weight of nitroglycerine.

3. An explosive composition comprising 12-15% of nitroglycerine, 50-80% inorganic explosive of the type of ammonium and sodium nitrate, and 10-20% of a low density carbonaceous ingredient consisting of clean-cut annual plant stalk, the interior cell walls of the particles of plant tissue

being substantially intact, said explosive composition having a density such that a 1 $\frac{1}{4}$ x8 inch cartridge weighs between seventy-five and ninety grams.

5 4. An explosive composition comprising a liquid sensitizing explosive, explosive salts, and a low density carbonaceous ingredient consisting of clean-cut particles of corn stalk, which have a density when compressed at 12 pounds per square  
10 inch between 0.075 and 0.150, and sorb at saturation not more than 2.00 times its weight of nitroglycerine.

5 5. In the art of making low density carbonaceous fillers for explosives, the process comprising, cutting annual plant stalks into particles while the stalks are moistened to their fiber-saturation point.

20 6. In the art of making low density carbonaceous fillers for explosives, the process comprising, cutting annual plant stalks into particles while the stalks are moistened to their fiber-saturation point, treating the cut particles with additional water, and drying.

25 7. In the art of making low density carbonaceous fillers for explosives, the process comprising, cutting annual plant stalks into particles while the stalks are moistened to their fiber-saturation point, treating the cut particles with additional water, and drying at temperatures between 100° and 150° C.

30 8. In the art of making low density carbonaceous fillers for explosives, the process comprising, cutting broom corn stalks into particles while the stalks are moistened to their fiber-saturation  
35 point, treating the cut particles with additional

water, and drying at temperatures between 100° and 150° C.

9. In the art of making low density carbonaceous fillers for explosives, the process comprising, cutting corn stalks into particles while the stalks are moistened to their fiber-saturation point, treating the cut particles with additional water, and drying at temperatures between 100° and 150° C.

10 10. In the art of making low density carbonaceous fillers for explosives, the process comprising, cutting sunflower stalks into particles while the stalks are moistened to their fiber-saturation point, treating the cut particles with additional water, and drying at temperatures between 100° and 150° C.

11. A low density carbonaceous filler for explosive compositions, comprising clean-cut particles of broom corn stalk of a fineness such as to pass an eight mesh and be retained on a thirty mesh  
20 screen, having a density between 0.075 and 0.150 when loaded under a pressure of twelve pounds per square inch, and capable of sorbing not more than 2.00 times their weight of nitroglycerin at saturation.

25 12. An explosive composition comprising, an explosive liquid nitric ester having a low density carbonaceous ingredient composed of clean-cut annual plant tissue having a density when compressed at 12 pounds per square inch of between  
30 0.075 and 0.150, said plant tissue being capable of sorbing, at saturation, not more than two times its weight of nitroglycerin.

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35