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PROCESS OF IMPREGNATING PLANT TISSUES WITH AMMONIUM NITRATE FOR EXPLOSIVE PURPOSES.

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To all whom it may concern:

Be it known that I, WILLIAM M. DEHN, a citizen of the United States, residing in Seattle, county of King and State of Washington, have invented certain new and useful Improvements in Processes of Impregnating Plant Tissues with Ammonium Nitrate for Explosive Purposes, of which the following is a full, clear, and exact description.

My invention relates to the process of manufacturing explosive mixtures of the type in which a combustible, derived from plant tissues, preferably wood meal, is intimately impregnated with the oxidant, ammonium nitrate. The chief object of my invention is to provide a powerful explosive of this type, which shall be safe to handle and can be manufactured at a minimum of cost. This is secured by my process which brings the two materials substantially into closest physical contact.

As early as 1867, Ohlsson and Norrbin secured patents on explosive compositions of ammonium nitrate and sawdust, etc. These and other inventors have combined the materials by different processes in varied proportions; however, no inventor has hitherto brought these materials into closest physical contact by methods of impregnation such as is revealed by this specification. The prior mixtures were insensitive towards detonation and were composed of materials existing in juxtaposition, and the ammonium nitrate did not saturate the cell walls and did not penetrate and substantially fill the cellular and tubular spaces of the plant tissues. When the ammonium nitrate is made thus substantially to fill the porous structures of the plant tissues, in concentrations approximating the stoichiometric ratios of the chemical equations:

$$C_{30}H_{50}O_{25}+30NH_4NO_3 \longrightarrow 30CO+85H_2O+30N_2 \text{ (74.8\% nitrate)}$$
$$C_{30}H_{50}O_{25}+60NH_4NO_3 \longrightarrow 30CO_2+145H_2O+60N_2 \text{ (85.5\% nitrate)}$$

important advantages can be secured.

A greater variety of forms and sizes of grains can be obtained, thus giving more varied control of velocities of detonation for different explosive purposes. Also, owing to increased sensitiveness of explosion, a greater range of such velocities of detonation can be secured by varying more widely the percentages of the materials used. The granular woody structure of the impregnated masses lessens the percentage of hygroscopy of the ammonium nitrate, prevents dead-pressing and melting of ammonium nitrate during manufacture and explosion, and insures complete explosion through the confinement and the combustible afforded the ammonium nitrate by the cell walls of the plant tissue. Owing to the elimination of air from the cells in these impregnated masses, greater densities of material can be obtained than with merely mingled mixtures, thus lessening dead spaces, securing greater certainty of detonation, and yielding greater disruptive power. Since, with very finely divided materials, explosive power, substantially equal to or greater than that of the nitrocelluloses, the nitrostarches, the ammonals, etc., can be obtained, more power per unit of cost can be be obtained with the impregnated composition than with the other high powered explosives. The closeness of contact of the ammonium nitrate and the cell walls, especially in the finely divided impregnated composition, approximates that of the nitro groups for cellulose in the nitro-celluloses. While thus approximating the intimacy of the latter, the impregnated masses conserve the exothermic energy lost during nitration of cellulose. When the impregnated plant tissue is a component in mixtures with trinitrotoluene, etc., the latter are not only rendered more sensitive to explosion but its percentage and the cost of the entire charge can be reduced very largely.

It is well known that, during the moment of detonation, certain fusible disruptive explosives, not properly ignited, ventilated or confined, tend to melt and pack to insensitive masses without exploding completely. This insensitiveness towards combustion, for example with large crystals of pure ammonium nitrate, trinitrotoluene, etc., on the one hand, can be overcome by increasing the charge of the igniter or by increasing the fineness and the confinement of the main charge. On the other hand, with impregnated masses described herein, the unfavorable tendencies are overcome by the toughness of grains, by their substantial nonfusible nature, by minute and momentary intercellular confinements of explosion gases during combustion, and by the critical temperatures resulting therefrom. It is obvious, therefore, with properly impregnated masses, larger granulations can be employed and greater progressiveness of explosion can result than with merely mingled masses of the same materials. It is chiefly the increased sensitiveness and power that give the impregnated masses their superior explosive qualities.

As an example of my process, I take wood meal or sawdust of 30–100 mesh fineness and soak it in water to obtain partial elimination of air and absorption of water in the porous structures. To the water and wet sawdust is added either immediately or later sufficient ammonium nitrate to yield the calculated desired composition. For example, four times as much ammonium nitrate as dry sawdust may be used. The temperature of the bath is first raised to the boiling point and then, through evaporation of water, can be carried up to the melting point of ammonium nitrate itself (169.6°) or preferably it is stopped at some lower point, practically at a point not to exceed 130°. If the temperature is carried up to the melting point of ammonium nitrate, carbonization, disintegration, nitration or combustion of the plant tissue will result. If the highest employed temperature is close to that of 100°, too much water will remain in the cooled product. This excess of water in the latter not only requires a long time for evaporation at the atmospheric or higher temperature, but also gives rise to less compact masses through creeping and efflorescence. However, since the products thus formed possess somewhat different explosive properties, and since the varied plant tissues possess different risistances to high temperatures, the process of impregnation practically will be carried out at that temperature between 100 and 169.6° which is substantially most efficient for yielding the products sought. Usually this temperature will not exceed 130°.

Briefly described, the process comprises the mixing of sawdust or other plant tissues with a saturated aqueous solution of ammonium nitrate and evaporating to dryness. As the mixture is heated, air is boiled out of the cells of the plant tissue and when the water is removed by evaporation, the cell and the cell walls are substantially filled with closely packed masses of ammonium nitrate, as may easily be observed under the microscope. The percentage contained in the cells depends not only upon the amount used but also upon the cellular structure of the plant tissue. For disruptive explosive purposes it is not practical or desirable to obtain a product containing all of the ammonium nitrate within the cells, for I have found that such products, though superior in power and sensitiveness of explosion to the merely mingled materials, are inferior to the products containing extracellular as well as intracellular ammonium nitrate. Explosion on both sides of the cell walls, therefore, favors progressiveness and completeness of detonation. However, when more intercellular ammonium nitrate than is obtained by one evaporation of water is desired, the caked mass can be treated with a little water a second time. Repeated adding of water and drying will eventually carry into the cellular structures substantially all of the ammonium nitrate. All of the water should finally be removed by evaporation at the atmospheric or elevated temperatures. Since the product is highly sensitive to efficient detonating charges, it possesses substantial tolerance to hygroscopic and incompletely evaporated water. The product is less susceptible to ordinary combustion than sawdust, hence the drying can safely and conveniently be carried out by steam heat properly applied. The caked mass is granulated or pulverized to the desired fineness and heated to remove the last traces of water. The product thus formed can be run directly into containers, shells, mines, etc., or can be pressed into forms suitable for dynamites, booster charges, etc.

The following table illustrates explosive advantages of the process of impregnation of plant tissues with ammonium nitrate. The grams of sand crushed were obtained by the use of one gram charges of the explosives ignited by No. 7 caps.

Impregnated.

Plant tissue.	%	Mesh.	% AN.	Final mesh.	Sand.
Fir sawdust	16	30–50	84	30	78
" "	16	100	84	100	90
" "	40	200	60	100	63
White ash bark	20	100	80	100	88
Nut shells	17	100	83	100	87
" "	20	30–90	80	30–90	76
Lycopodium	19	200	81	100	89
Wood charcoal	13	100	87	100	67
Starch	16	200	84	100	88
Average					87

Merely mixed.

Plant tissue.	%	Mesh.	Mesh AN.	Sand.
Fir sawdust	30	20–30	20–30	32
" "	20	20–30	20–30	26
" "	30	30–50	30–100	26
" "	20	30–50	30–100	57
" "	10	30–50	30–100	58
" "	30	30–90	30–100	42
" "	20	30–90	30–100	55
" "	10	30–90	30–100	56
Starch	16	200	100	71
Average				47

Here it is clearly demonstrated that nearly twice as much effective power is obtained with impregnated masses as with mingled masses of the same materials. It is certain that the differences are still greater when the explosive power of greater masses than one gram are measured, especially when exploded under conditions of imperfect confinement, inasmuch as any difference here represents a falling off of the explosive wave of the insensitive ammonium nitrate. The data of the table further demonstrates that different plant tissues give little variations of effective disruptive power, therefore, porous meal from any plant, containing not only wood and bark but also other plant materials, can be used for the manufacture of such impregnated ammonium nitrate explosives. Besides the materials mentioned in the table, cut straw and grasses, mealed roots and leaves, etc., have been found to yield highly impregnated masses possessing comparable explosive properties.

From the foregoing it is obvious that the applicant's invention does not depend upon the use of the tissues from a particular plant or upon the use of a particular tissue from any plant or upon a particular combination of the ingredients, but upon the physical condition of the explosive resulting from a substantial filling-up of the cells and the cell walls of said plant tissue with ammonium nitrate. Compositions of a seemingly analogous character, formed by mere mixing or mingling of materials and observed under the microscope to be substantially extracellular, segregated and unconsolidated, are not to be confused with the product obtained by my described simple process of intercellular impregnation. Applicant's invention involves the embedding of the main portion of the ammonium nitrate within the cells and cell walls of any plant tissue adapted to receive said ammonium nitrate and these consolidated grains may advantageously have a smaller portion of finely divided ammonium nitrate adhering to or admixed with the consolidated, impregnated grains, so that combustion and explosion may occur simultaneously on both sides of the cell walls. The appearance of some extracellular ammonium nitrate in my explosive must not lead to the confusion that the explosive is a simple mixture of ammonium nitrate with wood meal or other plant tissues. My composition not only differs from such in lessened porous structure, in deepened color, in greater hardness and explosive power, but also is observed under the microscope to be a mixture of a little extracellular ammonium nitrate in contact with consolidated masses which are composed of plant tissues holding intercellularly ammonium nitrate in the preponderating proportion.

When impregnated wood meal is combined with trinitrotoluene, picric acid or other explosives, the percentages of the latter in mixtures with ammonium nitrate can be reduced without loss of sensitiveness of explosion and with little or no loss of disruptive power.

	$\%NH_4NO_3$.	% T.N.T.	Sand crushed.
Impregnated wood meal.90%..	(75)	10	97
Ammonal. A1, 23.5%, C, 4.5%..	72	....	92
Amatol..........	55	45	102
Thunderite........flour, 4%..	92	4	67
Ammonium picrate.....60%..	40	....	78
Picric acid............100%..	....	....	87

It is evident from the above table that a great variety of useful explosive compositions can be prepared that will contain as the preponderating ingredient plant tissue impregnated with ammonium nitrate. The remainder of the composition can advantageously be nitrocellulose, nitroglycerine trinitrotoluene, or any of the other nitro aromatic explosive bases. All of these, involving as they do my process of manufacture of the impregnated ingredient, are construed to fall within the scope of my claims.

I claim:

1. The process of impregnating plant tissues with ammonium nitrate for explosive purposes, which comprises heating their mixture with water and evaporating the water.

2. The process of impregnating wood meal with ammonium nitrate for explosive purposes, which comprises heating their mixture with water and evaporating the water.

3. The process of impregnating plant tissues with ammonium nitrate for explosive purposes, which comprises heating a mixture of finely divided plant tissues, water and ammonium nitrate until substantially the cellular air and all the water are expelled and the plant cells and cell walls are filled with the preponderating proportion of the ammonium nitrate.

4. The process of impregnating wood meal with ammonium nitrate for explosive purposes, which comprises heating a mixture of wood meal, water and ammonium nitrate until substantially the cellular air and all the water are expelled and the woody cells and cell walls are filled with the preponderating proportion of the ammonium nitrate.

5. The process of impregnating plant tissues with ammonium nitrate for explosive purposes, which comprises heating to dryness at temperatures below 130° a mixture of plant tissue, water and ammonium nitrate.

6. The process of impregnating wood meal with ammonium nitrate for explosive purposes, which comprises heating to dryness at temperatures below 130°, a mixture of wood meal, water and ammonium nitrate.

7. The process of impregnating plant tissues with ammonium nitrate for explosive purposes, which comprises first boiling a mixture of plant tissues, water and ammo-

nium nitrate until the air is expelled from the plant cells and most of the water is evaporated from the mixture and then completing the evaporation of water and the substantial inclusion of the ammonium nitrate in the plant cells by drying at temperatures lower than said boiling temperature.

8. The process of impregnating wood meal with ammonium nitrate for explosive purposes, which comprises first boiling a mixture of wood meal, water and ammonium nitrate until the air is expelled from the plant cells and most of the water is evaporated from the mixture and then completing the evaporation of water and the substantial inclusion of the ammonium nitrate in the cells by drying at temperatures lower than said boiling temperature.

9. The process of impregnating plant tissues with ammonium nitrate for explosive purposes, which comprises heating plant tissues with a concentrated aqueous solution of ammonium nitrate, evaporating the water and grinding the cake resulting therefrom.

10. The process of impregnating wood meal with ammonium nitrate for explosive purposes, which comprises heating wood meal with a concentrated aqueous solution of ammonium nitrate, evaporating the water and grinding the cake resulting therefrom.

11. The process of impregnating plant tissues with ammonium nitrate for explosive purposes, which comprises forming a mixture of 15—35 parts of finely divided plant tissue and a concentrated aqueous solution of 100 parts of ammonium nitrate, heating and boiling this until the temperature practically becomes 130°, continuing the evaporation of water at lower temperatures, grinding the cake resulting and completing the evaporation of water.

12. The process of impregnating wood meal with ammonium nitrate for explosive purposes, which comprises forming a mixture of 15–35 parts of wood meal and a concentrated aqueous solution of 100 parts of ammonium nitrate, heating and boiling this until the temperature practically becomes 130°, continuing the evaporation of water at lower temperatures, grinding the cake resulting and completing the evaporation of water.

13. An explosive comprising plant tissues, impregnated with ammonium nitrate by the use of water solution thereof, substantially as described.

14. An explosive comprising wood meal, impregnated with ammonium nitrate by the use of water solution thereof, substantially as described.

15. An explosive comprising plant tissue, impregnated with ammonium nitrate, the latter in the preponderating proportion and substantially filling the cells and the cell walls of the former.

16. An explosive comprising wood meal, impregnated with ammonium nitrate, the latter in the preponderating proportion and substantially filling the cells and the cell walls of the former.

17. An explosive comprising a preponderating proportion of ammonium nitrate which is largely included within the cells of finely divided plant tissues.

18. An explosive comprising a preponderating proportion of ammonium nitrate which is largely included within the cells of wood meal.

19. An explosive comprising plant tissues and ammonium nitrate, the latter in both intercellular and extracellular relation to the former.

20. An explosive comprising wood meal and ammonium nitrate, the latter in both intercellular and extracellular relation to the former.

21. An explosive composed of 15–25% of plant, tissue, impregnated with 75–85% of ammonium nitrate.

22. An explosive composed of 15–25% of wood meal, impregnated with 75–85% of ammonium nitrate.

In testimony that I claim the foregoing, I hereto set my hand, this 12 day of April, 1921.

WILLIAM M. DEHN.