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IMPREGNATED MATERIAL AND METHOD OF IMPREGNATION

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My invention relates to an improvement in impregnated material and method of impregnating.

While my invention may be advantageously adapted to various arts, it is of particular advantage in connection with the production of explosives, more particularly of the type generally known as permissible explosives, in connection with which, for illustrative purposes, I will describe my invention in detail.

In the manufacture of explosives, and more particularly of explosives of the permissible type which are used in the mining of coal, it is desirable that the explosive be of low density in order that when used it will not operate to shatter the coal with the production of relatively small pieces of slack rather than relatively large lumps. Desirably the permissible explosive should have a density such that in operation it will have an effect comparable to that of black powder.

The permissible explosive, as is well known, is composed of a mixture of an explosive salt, as for example, ammonium nitrate, an oxidizing salt, as for example, sodium nitrate, carbonaceous material, as for example, wood pulp, or a low density material as bagasse, or the like, balsa wood, bongo wood, or the like, and a sensitizer such as nitroglycerine, or other liquid high explosive.

In the manufacture of permissible explosives heretofore, since the density of the explosive and oxidizing salts and of the sensitizer are more or less fixed, it has been attempted to obtain the feature of desirable low density of the composition by the use of a carbonaceous ingredient of the lowest density obtainable, but such attempts have not been productive of results comparable to that desired, since if a low density carbonaceous material is used in sufficient quantity it will very quickly absorb the sensitizer to a degree such that the explosive will be so insensitive to detonation as to be useless for all practical purposes. Heretofore it has also been suggested that a sufficient quantity of low density carbonaceous material be used to obtain the desired density of the explosive and that the absorptiveness of the carbonaceous material be negatived by impregnating into the carbonaceous material a portion of the salt ingredients of the explosive. With the use of impregnated carbonaceous material as impregnated pulp, bagasse, or low density woods, as balsa wood, bongo wood, or the like, an explosive of desirable low density, as for example, one having a cartridge count of five hundred or more 8″ x 1¼″ cartridges per hundred pounds, may be produced, as disclosed in the United States patents granted to me, No. 1,671,792, dated May 29, 1928, and No. 1,671,793, dated May 29, 1928.

Now, in accordance with my present invention, I have found that the impregnation of various materials of low density, such as low density pulp, low density woods, as balsa wood, bongo wood, and the like, may be efficiently and economically carried out through the medium of a fused mixture of salts in eutectic proportions, or a mixture of salts in any proportion where fusion of a portion of the mixture will occur at a eutectic temperature, such mixtures including where the impregnated material is for use in explosives, explosive and oxidizing salts forming ingredients of the explosive.

In the practical adaptation of my invention for the impregnation of pulp for use in explosives, desirably a eutectic mixture of salts of which ammonium nitrate is the preponderating component, having a eutectic temperature as close to 100° C. as possible, and in any event below a dangerous temperature in connection with the salts used is formed. To the eutectic mixture may be added, if desired, a small quantity of a salt containing water of crystallization, or a small quantity of free water in order to accelerate melting and in the case of a salt containing water of crystallization to delay crystallization, though the inclusion of such is not essential. In effecting impregnation of the desired material in accordance with my invention, the eutectic mixture is made liquid by fusing and the liquid mixture absorbed into the carbonaceous material, which after absorption is cooled to a temperature

below the eutectic temperature and at which it will crystallize.

It will now be understood that in accordance with my invention I effect the impregnation of a material to be impregnated through the medium of a eutectic mixture of salts, or, in other words, through the medium of a mixture of salts in such proportion that the composition of the melt is the same as the composition of the solid separating from it, the actual impregnation being effected with the eutectic mixture in a fused condition brought about by heating to, or slightly above, its eutectic temperature, the temperature being desirably maintained until sufficient absorption of the melt by the material to be impregnated has occurred. When desirable absorption has occurred the mass is cooled to a temperature below the eutectic temperature of the mixture of salts with the result that the salts crystallize, with the production of a desirably impregnated product and one which is anhydrous or substantially anhydrous. Alternatively, in accordance with my invention I may effect the impregnation through the medium of a mixture of salts in any proportion, fusion of a portion of the mixture being effected by heating to the temperature at which a eutectic mixture of the salts will be fused.

While in the practical adaptation of my invention where a eutectic mixture is used, the impregnation may be effected through the medium of a eutectic mixture as such, under certain circumstances a small amount of free water may be added to the mixture in order to facilitate fusion, or there may be added to the eutectic mixture a hydrated salt (i. e. one containing water of crystallization) which will have the effect of delaying crystallization and hence of insuring thorough impregnation of the material to be impregnated.

By way of illustrating the practical adaptation of my invention, more particularly in connection with the use of a eutectic mixture, for example, a eutectic mixture involving the system ammonium nitrate-sodium nitrate-ammonium chloride, the ammonium nitrate being in preponderating proportion, or such a system involving the use of both sodium nitrate and potassium nitrate may be used. To such a system a small amount of free water may be added, which, however, will substantially disappear in the impregnating operation at the eutectic temperature of the mixture leaving the product substantially anhydrous, or a small amount of a hydrated salt such as, for example, magnesium nitrate hexahydrate, calcium nitrate tetrahydrate, or the like, may be added with or without the addition of free water.

Eutectic mixtures involving the systems ammonium nitrate-sodium nitrate-sodium chloride or ammonium chloride may be made having a eutectic temperature of 112.5 C., and with the addition of a small amount of free water, or a hydrated salt, the temperature of complete solution may be lowered to less than 105° C.

The practical adaptation of my invention to production of a eutectic mixture involving the system ammonium nitrate-sodium nitrate-sodium chloride, or ammonium chloride, adaptable, for example, more especially for the production of impregnated material for use as an ingredient of permissible explosives, may be illustrated by the formulæ in the following table:

Table A

	No. 1		No. 2
Ammonium nitrate	75.8	Ammonium nitrate	83.6
Ammonium chloride	5.2	Sodium chloride	5.7
Sodium nitrate	19.0	Sodium nitrate	10.7
Eutectic temp. ° C	112.5	Eutectic temp. ° C	112.5

In connection with the above formulæ, it will be observed that Formula No. 2 differs from Formula No. 1 in the use of sodium chloride as used in place of ammonium chloride. However, it will be noted that sodium chloride will react with ammonium nitrate to form ammonium chloride and sodium nitrate and hence that Formula No. 2 is in effect equivalent to Formula No. 1 in that the same eutectic, i. e. having a eutectic temperature of 112.5° C., is produced through reciprocal salt combinations. Thus, it will be noted that the same eutectic may be produced with the use in one instance of ammonium chloride and in another instance with the use of sodium chloride.

Under practical conditions using sodium chloride, when the ammonium nitrate and sodium chloride are fused together the salts undergo double decomposition forming some ammonium chloride and some sodium nitrate. Thus, under Formula No. 2 sufficient excess of ammonium nitrate and sufficient sodium chloride are provided to produce the required ammonium chloride and sodium nitrate to form with the sodium nitrate originally provided and the undecomposed ammonium nitrate the eutectic mixture formed under Formula No. 1.

Impregnation of any desired material, such as wood pulp, balsa wood, bongo wood, bagasse, or other low density absorptive material when the product is desired for use as an ingredient of explosives, may be effected with mixtures prepared according to either of the above formulae by subjecting the material to be impregnated to the mixture after fusing at or above the eutectic temperature, which desirably is maintained until the material is saturated, after which mere cooling of the mass, which effects solidification of the salts, completes the product.

The formulæ shown in the following table

more specifically illustrate the application of my invention to the production of an impregnated product, with the use of eutectic mixtures involving the system ammonium nitrate-sodium or ammonium chloride-sodium nitrate, with and without the addition of a small amount of free water, and of a small amount of free water and of a hydrated salt:

Table B

	No. 1	No. 2	No. 3	No. 4
Ammonium nitrate	57.0	55.6	52.4	59.7
Ammonium chloride	3.8	3.7	3.5	-------
Sodium nitrate	14.2	13.8	13.1	7.6
Sodium chloride	-------	-------	-------	4.1
Magnesium nitrate hexahydrate	-------	1.9	6.0	6.2
Balsa pulp	25.0	25.0	25.0	22.4
Free water (basis total salts)	2.5	2.5	-------	-------

The eutectic salt compositions, as shown by Table A, are maintained throughout. If free water and hydrated salts are omitted from any of the formulæ given in Table B, the resultant is a eutectic mixture of salts melting at 112.5° C. The melting point of these salt mixtures when free water and/or a hydrated salt is added is about 100° C.

In the preparation of impregnated material in accordance with the formulæ of the above Table B the free water which is included will be substantially lost with the production of a product containing less than 0.5% of moisture, or one which is substantially anhydrous. In the case of Formula No. 1, when no free water is added, the eutectic temperatures will be 112.5° C. while in the case of formulæ Nos. 2, 3 and 4, which include free water and a hydrated salt (No. 2) or a hydrated salt (Nos. 3 and 4), the hydrated salt, as magnesium nitrate hexahydrate, which latter is specially advantageous in effecting a lag or delay in crystallization of the mixture, the temperature of complete solution will be reduced to about 100° C.

As has been indicated, in accordance with my invention I may effect impregnation by the use of a mixture of salts in any proportion, a portion of the mixture being fused at a eutectic temperature for the system of salts involved. Thus, for example, a mixture of ammonium nitrate, sodium or ammonium chloride and sodium or potassium nitrate, in any proportions, is fused in part by heating to a temperature of about 112.5° C., the eutectic temperature of such system of salts, or at a lower temperature, say about 105° C. where a small amount of free water or a hydrated salt is added to the mixture as described in connection with the eutectic mixture. The fusion of a part of the mixture will occur at the eutectic temperature for the system of salts involved since the salts will fuse in eutectic proportions irrespective of the presence of an excess of one or more of the salt ingredients of the mixture. In effecting impregnation the material to be impregnated is subjected to the mixture fused in part, which consists of liquid or fused salt carrying solid salt in suspension. The fused or liquid portion of the mixture is absorbed by the material to be impregnated and on cooling solidifies, while the solid portion of the mixture becomes mechanically admixed with the impregnated material and may be to a degree carried into the cellular structure of the material by the liquid portion.

The impregnated material produced in accordance with my invention, when the system ammonium nitrate-ammonium chloride-sodium nitrate, or sodium nitrate-potassium nitrate, and more especially when the material impregnated is of relatively low density and relatively high absorptive capacity as, for example, wood pulp, balsa wood, bongo wood, bagasse, etc., etc., is, as has been indicated, adaptable for the production of permissible explosives having such desirable characteristics as high cartridge count, resistance to setting, retention of sensitivity, etc., etc. to a maximum degree. Permissible explosives involving the impregnated material according to my invention, when the material is impregnated through the medium of a eutectic mixture of salts, or through the medium of a partially fused mixture of salts in any proportion, with and without the use of free water, or of a hydrated salt may be made up according to the following formulæ:

Table C

	No. 1	No. 2	No. 3	No. 4
"R" nitroglycerin	12.0	12.0	12.0	12.0
Impregnated material	68.0	48.0	64.0	71.0
No. 1 ammonium nitrate	12.0	40.0	14.5	5.4
Sodium nitrate	8.0	-------	9.5	11.6

From the above Table C it will be noted that the explosive may contain either the ammonium or sodium nitrate ingredients entirely as present in the impregnated material, or proportions of such may be mixed with the material. The powder in accordance with the formulæ of the above Table C will have the ultimate composition and characteristics as shown in the following table D:

Table D

	No. 1	No. 2	No. 3	No. 4
"R" nitroglycerin	12.0	12.0	12.0	12.0
Ammonium nitrate	50.7	66.7	48.1	47.8
Ammonium chloride	2.6	1.8	2.2	-------
Sodium nitrate	17.7	6.6	17.9	17.0
Sodium chloride	-------	-------	-------	2.9
Magnesium nitrate hexahydrate	-------	0.9	3.8	4.4
Balsa pulp	17.0	12.0	16.0	15.9
Count (1¼ x 8) 100 lbs	540	456	501	553
Sensitivity—normal	10″	16″	16″	10″
Sensitivity—24 hrs. at 100° F	10″	14″	16″	10″
Sensitivity—120 hrs. at 100° F	8″	14″	14″	10″
Velocity—m/s	1764	2212	2090	1922

It will now be understood that in accord-

ance with my invention, from the broad standpoint, a method of effecting the impregnation of materials is afforded which involves impregnation through the medium of a eutectic mixture of salts at or above the eutectic temperature, or of a mixture of salts fused in part, followed by cooling to effect crystallization or solidification of the salts, and the production of an impregnated material which is anhydrous or substantially anhydrous. More particularly, in accordance with my invention the addition to the eutectic mixture of a small amount of free water and of a hydrated salt is contemplated for the purpose of lowering the temperature of complete solution and/or retarding or delaying recrystallization or solidification of the salts on cooling. Still more particularly, my invention involves the impregnation of absorptive carbonaceous material with a eutectic mixture involving the system ammonium nitrate-ammonium chloride-sodium nitrate, with the ammonium nitrate in predominating proportion, and to which may be added a small amount of free water and/or a hydrated salt, with the production of a product adaptable for use as an ingredient of explosives. My invention also contemplates explosives involving as an ingredient impregnated material in accordance with my invention.

As a result of my invention, it will be noted that a simple and efficient method is provided for effecting the impregnation of material at low temperatures, as below 115° C., thus enabling the impregnation of explosive salts, such as ammonium nitrate, and the like, without danger and at the same time enabling impregnation without the use of free water, or with the use of so small an amount of free water as to leave less than 0.5% in the product, thus enabling the production of an anhydrous or substantially anhydrous product. As a more specific result of my invention, a simple and efficient method for the production of impregnated material for use in explosives is provided and which, with the use of low density material, such as low density wood pulp, bagasse, balsa wood, bongo wood and the like, will enable the production of explosives having high cartridge count, as above five hundred standard $1\frac{1}{4}'' \times 8''$ cartridges per 100 pounds, having a retention of sensitivity, resistance to setting, and desirable power or explosive effect.

It will also be understood that I do not intend that my invention shall be limited to the use of any particular salts, nor to any particular material to be impregnated, nor to any particular uses of the impregnated material.

What I claim and desire to protect by Letters Patent is:

1. The method of producing impregnated material which includes admixing a material to be impregnated with a mixture of salts fused at about a eutectic temperature for the system of salts involved and cooling to effect solidification of the salts.

2. The method of producing impregnated material which includes admixing a material to be impregnated with a mixture of ammonium nitrate, ammonium chloride and sodium nitrate fused at about a eutectic temperature and cooling to effect solidification of the salts.

3. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of ammonium nitrate in preponderating proportion, ammonium chloride, and sodium nitrate in a fused state and cooling to effect solidification of the salts.

4. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of salts and free water, the eutectic mixture being in a fused state.

5. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of ammonium nitrate in preponderating proportion, ammonium chloride, and sodium nitrate in a fused state, the melt containing free water, and cooling to effect solidification of the salts.

6. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of salts, with a hydrated salt added, in a fused state and cooling to effect solidification of the salts.

7. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of ammonium nitrate, ammonium chloride, and sodium nitrate, with a hydrated salt added, in a fused condition and cooling to effect solidification of the salts.

8. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of ammonium nitrate in preponderating proportion, ammonium chloride and sodium nitrate, with magnesium nitrate added, in a fused state and cooling to effect solidification of the salts.

9. The method of producing impregnated material which includes admixing a material to be impregnated with a eutectic mixture of ammonium nitrate, ammonium chloride and sodium nitrate, with a hydrated salt added, in a fused state, the melt containing free water, and cooling to effect solidification of the salts.

10. As a new product absorptive material impregnated with a eutectic mixture of salts.

11. As a new product absorptive material impregnated with a eutectic mixture of salts

including ammonium nitrate, ammonium chloride and sodium nitrate.

12. As a new product absorptive material impregnated with a mixture of salts including ammonium nitrate in preponderating proportion, ammonium chloride and sodium nitrate.

13. As a new product absorptive material impregnated with a mixture of salts including ammonium nitrate, ammonium chloride, and sodium nitrate, with a hydrated salt added.

14. As a new product absorptive material impregnated with a mixture of salts including ammonium nitrate in preponderating proportion, ammonium chloride and sodium nitrate, with magnesium nitrate added.

15. A a new product absorptive material impregnated with a mixture of salts including ammonium nitrate, ammonium chloride and sodium nitrate, the product containing less than 0.5% free moisture.

16. An explosive including in admixture an explosive ingredient and carbonaceous material impregated with a eutectic mixture of salts one of which is an explosive salt.

17. An explosive including in admixing an explosive ingredient and carbonaceous material impregnated with a eutectic mixture of salts one of which is ammonium nitrate.

18. An explosive including in admixture an explosive ingredient and carbonaceous material impregnated with a mixture of ammonium nitrate, ammonium chloride, and sodium nitrate in such proportions that the mixture will fuse at a temperature less than 115° C.

19. An explosive including in admixture an explosive ingredient and carbonaceous material impregnated with a eutectic mixture of salts one of which is ammonium nitrate, the impregnated material containing less than 0.5% of free moisture.

20. An explosive including in admixture an explosive ingredient and carbonaceous material impregnated with a mixture of ammonium nitrate, ammonium chloride and sodium nitrate, with a hydrated salt added.

21. The method of producing impregnated material which includes forming a eutectic mixture of salts involving when in a fused state the system ammonium nitrate-ammonium chloride-sodium nitrate, effecting the impregnation of an absorptive material with the mixture in a fused state and cooling the impregnated material.

22. The method of producing impregnated material which includes forming a eutectic mixture of salts involving when in a fused state the system ammonium nitrate-ammonium chloride-sodium nitrate, fusing the salts forming the mixture in the presence of free water, effecting the impregnation of an absorptive material with the mixture in a fused state and cooling the impregnated material.

23. The method of producing impregnated material which includes forming a eutectic mixture of salts involving when in a fused state the system ammonium nitrate-ammonium chloride-sodium nitrate, fusing the salts forming the mixture in the presence of a hydrated salt, effecting the impregnation of an absorptive material with the mixture in a fused state and cooling the impregnated material.

24. The method of producing impregnated material which includes forming a mixture of salts including reciprocal salts which will produce when fused a eutectic involving the system ammonium nitrate-ammonium chloride-sodium nitrate, effecting the impregnation of an absorptive material with the mixture in a fused state and cooling the impregnated material.

25. The method of producing impregnated material which includes forming a mixture of salts including ammonium nitrate, sodium chloride, and sodium nitrate, the ammonium nitrate and sodium chloride being in quantity sufficient so that on fusion of the mixture a eutectic involving the system ammonium nitrate-ammonium chloride-sodium nitrate will be formed, through double decomposition of sodium chloride and ammonium nitrate, effecting the impregnation of an absorptive material with the mixture in a fused state and cooling the impregnated material.

26. An explosive including ammonium nitrate admixed with a carbonaceous material impregnated with a eutectic mixture of salts one of which is ammonium nitrate.

27. An explosive including a sensitizer and carbonaceous material impregnated with a eutectic mixture of salts one of which is ammonium nitrate.

28. The method of producing impregnated material which includes forming a mixture of salts which is fusible at a temperature of about 112.5° C. or below and which contains an explosive salt, effecting the impregnation of absorptive material with the mixture in a fused state and cooling the impregnated material.

In testimony of which invention, I have hereunto set my hand at Kenvil, N. J., on this 1st day of October, 1929.

BUREN I. STOOPS.