

## UNITED STATES PATENT OFFICE

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## EXPLOSIVE

No Drawing.

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This invention relates to improvements in explosives. The object of the invention is to produce explosives having points of merit not possessed by similar explosives manufactured before the present invention. Another feature of the invention resides in the increased degree of safety in the compounding of same.

The invention applies to two kinds of explosives, viz:

1. Progressive combustion, or propellant explosives.

2. Detonating, or blasting explosives.

In the manufacture of propellants, it is customary in the prior art to employ nitrocellulose as such, made into a colloid by means of a suitable solvent such as ether-alcohol mixture or its equivalent, and this colloid is then forced through suitable dies to form strips, cords or grains. After which process, the strips, etc., are placed in a suitable oven, preferably containing a vapor of the solvent with which the colloid is prepared, and the solvent from the colloid is slowly removed, until there is little or none left in the mass. This drying is conducted very slowly at a low temperature. This type of explosive propellant is called monobase, there being only one form of explosive employed in its preparation.

Two of the most important requirements in a propellant are:

1. Chemical stability.

2. Absence of brittleness.

Chemical stability ensures the safety in storage of the explosive, and absence of brittleness ensures the uniformity of ballistics when the explosive is fired in the gun.

Another type of propellant is the duo-base, this is composed of a solid explosive, such as nitrocellulose, and a liquid explosive (nitroglycerine being commonly employed), usually colloidized by means of acetone or its equivalent. The resulting colloid being forced

through dies to form strips, etc., exactly as is done in the case of the mono-base propellant.

In the usual practice of the art, it is common practice to add other materials both in making the monobase and duo-base explosives.

There are several disadvantages in the use of common nitrocellulose, among which may be mentioned the comparative difficulty in getting this product uniformly stable, the relative trouble incurred in its manufacture, and the danger incident to handling a product of its characteristic physical condition. The present invention relates to the substitution of nitrated vegetable parchment in place of ordinarily nitrocellulose in the preparation of the propellant explosives above mentioned. Nitrated vegetable parchment is easy to manufacture, both from the view point of nitrating and stabilizing, and in the shredded condition is infinitely more simple to make and to handle in the factory, in addition to being much safer through all the stages of manufacture. From experiments conducted with the colloid prepared from nitrated vegetable parchment, it appears that much less solvent is required, and consequently, the trouble with shrinkage experienced with nitrocellulose colloids on drying, are very greatly reduced when nitrated vegetable parchment is used. With this latter, also the dried product is very tough, a property as previously pointed out, of great value.

A nitrated vegetable parchment can be made in accordance with the process disclosed in copending patent applications 132,906 issued December 16, 1930, Patent No. 1,785,050 and 132,907 filed August 1, 1926.

The said process consists essentially in that a good grade of pulp, preferably alpha fibre pulp, is first made into sheets or water-leaf, of about the thickness of 20 to 25 pound paper. These sheets are then parchmentized by the usual method, for example by immers-

ing the same in sulphuric acid of about 70 to 71% concentration, at preferably not above 60° F., the immersion being for not more than a few seconds, say about three seconds, after which the sheets are washed and dried.

Before nitration the sheets are preferably shredded into relatively small pieces of a wavy character in order that they may be nitrated in bulk with free access of the nitrating acids to every and each piece of vegetable parchment. These wavy pieces of vegetable parchment may be about  $\frac{1}{4}$  inch wide by 3 inches long. The size however can vary within very wide limits and is unimportant. The nitration is effected by means of a mixture of nitric and sulphuric acids of the desired proportions and strength, and at about 30° to 40° C. Such acid mixture for nitration may be about 60% sulphuric acid, 21 to 22% nitric acid and 18 to 19% water.

The nitration can be conducted in centrifugal machines or pots but preferably the displacement process is used, the shredded parchmentized paper being first made up into a bale of such a size as to fit loosely into the nitrating pan, and the acid may be run in from the bottom of the pan, the bale of material being held down by a suitable perforated acid-resisting plate placed on top of the same. The bale of shredded parchmentized paper may be bound with suitable tie wires such as lead coated iron wires or aluminum wires, before being put into the nitrating pan. The apparent weight of the bale may be 4 pounds per cubic foot.

After the nitration is completed, and if the displacement process is to be followed (which is far preferable to any other), then water is allowed to flow gently on to the surface of the acids, and at the same time the acids are being drawn off at the bottom of the nitrating vessel, and preferably at the same rate as the water is flowing on to the surface of the acids. The outflowing acids are allowed to flow till such time as there is a decided drop in the gravity of such acids, when a "cut" is made, and the two strengths of acids kept apart. When a second decided drop of gravity appears, then the balance of the acid is considered as waste and is run into the drain.

At this juncture the nitrated vegetable parchment will be found to contain very little acid, just enough to render it sour to the taste. It is now placed in the boiling vats, and given a good boiling in water for several hours, after this the water is changed and another boiling given and this process continued till such time as the nitrated-product is not only free from acid but will stand the several heat tests usually applied to nitrated products. The addition of a very small quantity of ammonium hydroxide (just alkaline to litmus) during the boiling process will

hasten the stabilization of the nitrated parchment. The product can then be dried.

The nitrated vegetable parchment so produced can be bleached if desired, say before the final drying, with an alkaline hypochlorite solution.

This product is far more uniform than any nitrated cotton or nitrated wood pulp obtainable by the older methods, and possesses better solubility, giving more concentrated solutions, and is more stable than any ordinary nitrocellulose.

As an ingredient of duo-base explosive propellant, nitrated vegetable parchment has distinct merit. A propellant of this class if prepared with nitrocellulose and nitroglycerine, using a common solvent usually acetone, the percentage of nitrocellulose in the composition cannot exceed a certain limit, otherwise the resulting finished grain, strip, or cord is too hard and brittle, a condition fatal to the quality of the propellant. If nitro-vegetable parchment is used, however, substantially larger percentages of this substance may be employed in the mass without developing such brittleness. This is very desirable for some propellants, particularly those in which the temperature of combustion is required to be low, and the normal gas volume high. Again, as in the case of the mono-base propellant, the amount of solvent required for colloiding is less than is required in the case of nitrocellulose.

In the most modern explosive propellant, di-nitro-di-glycol is used to form a duo-base explosive, as a substitute for nitro-glycerine. This substance has a powerful solvent action on nitro-vegetable parchment and very little common solvent is needed to form the colloid.

Much more nitro-vegetable parchment can be incorporated with the liquid explosive, than is possible with common nitrocellulose, to form a tough product. This is due, apparently, to the different natural physical states of the materials.

It is obvious that this is a great advantage, for the reason that if a nitrogen percentage of the finished propellant is required to be around 12.59 (which is the case in many standard propellants) more flexibility is given to the product if a large proportion of the nitro-vegetable parchment can be employed with the di-nitro-di-glycol (which has a nitrogen content of 14.7%).

This invention also embraces the substitution of nitrated vegetable parchment for nitrocellulose in the preparation of detonating or blasting explosives. In the preparation of this type of explosive, nitro-glycerine, nitroglycol, di-nitro-di-glycol and often some nitrohydrocarbons are used, these being gelatinized by means of nitrocellulose, and this mass incorporated with a suitable "dope", composed for example, of sodium nitrate,

wood pulp, and other ingredients of a character depending upon the uses for which the finished explosive is destined. If high grade, and stable (by this term reference is made to

Di-nitro-di-glycol, and the methods of making same are fully described in my co-pending application 323,583 filed December 3, 1928.

The nitrogen content of a mono-base propellant explosive, prepared in accordance with the present invention may vary between about 12% and about 12.6% of nitrogen. A duo-base propellant explosive prepared in accordance with the present invention may contain around 12% to 15.5% of nitrogen. In addition to the above ranges, certain explosives are made for saluting purposes or for maneuvering purposes, which may contain about 11.5% of nitrogen.

*Example 3.* In this example reference is made to a relatively high nitrogen duo-base propellant powder.

Tri-nitro-glycerine, or di-nitro-ethylene glycol, or a mixture of both is mixed with the nitrated vegetable parchment, adding sufficient of a common solvent such as acetone or its equivalent, to bring into perfect solution the ingredients, a little vaseline may be added to assist in lubrication of the gun barrel.

The proportions of the ingredients for general ordnance purposes may be:

|                                   |          |  |
|-----------------------------------|----------|--|
| Tri-nitro glycerine -----         | 40 parts |  |
| Nitrated vegetable parchment ---- | 55 parts |  |
| Vaseline -----                    | 5 parts  |  |

This paste is then extruded through dies of such shape as to give the desired shape of cords, strips, etc., after which process the solvent is evaporated and if desired, recovered.

*Example 4.* Detonating explosive.

In this example reference is made to a composition of nitrated vegetable parchment and tri-nitro-glycerine, or ethylene glycol di-nitrate or a mixture of these and to which tri-nitro-glycerine or ethylene glycol di-nitrate or a mixture of both may be added, di-ethylene glycol di-nitrate in desired quantity, which will reduce the freezing point of the mixture.

As this substance is destined to be one of a detonating nature, and therefore should have the maximum velocity of detonation to function best as a blasting explosive, the proportion of nitrated vegetable parchment must not be too high, otherwise the product will cease to be in the detonating class, and will only be a "progressive combustion powder."

The best composition should be as follows:

|                                   |          |  |
|-----------------------------------|----------|--|
| Tri-nitro glycerine -----         | 97 parts |  |
| Nitrated vegetable parchment ---- | 3 parts  |  |

or in case of mixtures with ethylene glycol di-nitrate as follows. Examples of detonating mixtures containing nitro-vegetable parchment, to be incorporated with "dopes":

|                                   |          |  |
|-----------------------------------|----------|--|
| Tri-nitro glycerine -----         | 97 parts |  |
| Nitrated vegetable parchment ---- | 3 parts  |  |

stability as determined by the standard government heat tests) nitro-cellulose be used in such compositions, then it is quite necessary to employ heat to cause the nitrocellulose to dissolve completely, and the application of heat to such a compound is in the highest degree hazardous. If, on the other hand, nitro-vegetable parchment is employed, such solution of the solid may be attained in the cold, namely at normal room temperature. In addition to this very valuable feature, using nitro-vegetable parchment, there is an entire absence of explosive dust, which is unavoidable when using nitrocellulose and which would add to the danger of explosion in preparation and storage.

The use of nitro-vegetable parchment, therefore, is not only a great advantage from the standpoint of an actual improvement in the explosive, but it is a safeguard to life and property.

The following examples are given for the purpose of illustration, it being understood that the invention is in no wise restricted to these examples.

*Example 1.* In this example a low nitrogen mono-base explosive is referred to. Nitrated vegetable parchment containing say 12% of nitrogen is dissolved in ether and alcohol mixture to form a saturated solution.

This is then subjected to extrusion under heavy hydraulic pressure, to form strips, cords and the like. These are dried in a suitable drying chamber or oven, and the volatile solvent mixture can be recovered if desired.

The drying may continue until about 0.1% of the solvent remains. The product is then cut into the desired sizes.

*Example 2.* In this example reference is made to a relatively low nitrogen duo-base powder. Di-nitro-di-glycol containing say 14.25% of nitrogen is mixed with nitrated vegetable parchment containing say 12.5% of nitrogen, in equal amount. Only sufficient solvent will be used to form a paste, for the reason that di-nitro-di-glycol is itself a good solvent especially for the nitrated vegetable parchment. The solvent is then evaporated as in the preceding example, and recovered if desired, and the operations are conducted as in the previous example. By increasing the amount of di-nitro-di-glycol the percentage of nitrogen in the explosive can be increased somewhat, since the di-nitro-di-glycol contains a larger percentage of nitrogen than the above mentioned nitrated vegetable parchment. It will be understood that the percentage of nitrogen in the final product can be adjusted by various other means such as the addition of high nitro-bodies or bodies not containing any nitrogen.

|    |                                    |          |
|----|------------------------------------|----------|
|    | Ethylene glycol di-nitrate.....    | 95 parts |
|    | Nitrated vegetable parchment.....  | 5 parts  |
|    | Tri-nitro glycerine .....          | 80 parts |
|    | Ethylene glycol di-nitrate.....    | 15 parts |
| 5  | Nitrated vegetable parchment.....  | 5 parts  |
|    | Tri-nitro-glycerine .....          | 92 parts |
|    | Di-ethylene glycol di-nitrate..... | 3 parts  |
|    | Nitrated vegetable parchment.....  | 5 parts  |
| 10 | Tri-nitro-glycerine .....          | 71 parts |
|    | Di-ethylene glycol di-nitrate..... | 5 parts  |
|    | Ethylene glycol di-nitrate.....    | 20 parts |
|    | Nitrated vegetable parchment.....  | 4 parts  |

15 The di-ethylene glycol di-nitrate is employed to reduce the freezing point of the mixture and also to reduce the sensitiveness of the nitroglycerine to shock or friction without reducing its sensitiveness to detonation (which happens if a nitro hydrocarbon is used).

20 Another very useful composition, which is sufficiently solid to be used as such, without the admixture of "dope" of any kind is the following:

|    |                                   |          |
|----|-----------------------------------|----------|
| 25 | Tri-nitro glycerine .....         | 93 parts |
|    | Nitrated vegetable parchment..... | 7 parts  |

30 or some of the nitro-glycerine may be substituted by ethylene glycol-di-nitrate, or di-ethylene glycol di-nitrate or mixtures of these.

I claim:—

35 1. A process which comprises colloidizing and drying a material comprising nitrated vegetable parchment, to produce an explosive.

40 2. A process which comprises colloidizing and drying a mixture including a nitrated normally liquid polyatomic alcohol and nitrated vegetable parchment, to produce an explosive.

45 3. A process which comprises colloidizing and drying a mixture including di-nitro-di-glycol and nitrated vegetable parchment, to produce an explosive.

4. A colloidized explosive containing nitrated vegetable parchment.

50 5. A colloidized explosive containing nitrated vegetable parchment and di-nitro-di-glycol.

In testimony whereof I affix my signature.

ARTHUR HOUGH.

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