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PROCESS OF MAKING NITROCELLULOSE POWDERS

No Drawing. Continuation of application Serial No. 589,722, filed September 21, 1922. This application filed April 11, 1927. Serial No. 183,013.

This invention relates to explosives and more particularly to nitrocellulose powders. This application is a continuation of application Ser. No. 589,722, filed September 21, 1922.

One of the objects of this invention is to develop a process for producing the explosive in a simple and economical manner.

Another object is to provide a process whereby the ballistic properties of the explosive can be closely controlled so as to obtain a finished product having the desired properties for the specific uses intended.

Another object is to provide a process in which the necessity of solvents is obviated.

Another object is to provide an explosive and more particularly a propellent powder having improved characteristics and properties so as to adapt the same for the specific uses intended.

Further objects will appear from the detailed description in which will be described a number of embodiments of this invention; it will be understood however that the described embodiments are simply for illustrative purposes since this invention is susceptible of various other embodiments.

Generally stated, in accordance with this invention, the nitrated cellulose is pulped to a consistency so as to enable it to be felted. The pulp nitrocellulose is gathered or felted into a sheet. This sheet is then subjected to wringing, drying and compressing operations so as to free the same of water and reduce it to the required dense state in order to adapt it for the uses for which it is intended.

The apparatus employed for the carrying out of the process embodying this invention may, generally stated, be the same as now used in the manufacture of paper; accordingly in the description to follow the elements and machines of a paper making plant will be referred to by their names as commonly used in the paper making industry. From a process standpoint however, there are certain differences in essential operation which must be clearly understood in order to enable one skilled in the art to practice the process embodying this invention.

The raw material forming the basis of the final product is raw cellulose or cotton as ordinarily used in the manufacture of nitrocellulose. It is not necessary to describe the preliminary treatment, since that is well known to those skilled in the art, consisting as it does of purifying the raw cellulose, nitrating the purified cellulose and purifying the nitrated cellulose. Further detailed description can, therefore, begin at the point where the cellulose has been nitrated and purified and is ready to be pulped.

The pulping operation is performed as a beating operation; this is an important procedure as it is by means of this operation that one of the most important ballistic properties, that is the rate of combustion of the finished powder, is controlled. The beating operation is carried out in any of the various types of beaters such as are used in the paper industry, examples of which are the Hollander, Jordan, Miller, Downingtown, etc., types. One of the functions of the beating operation is to cut the fiber to the proper length for felting; another important function, however, is to convert a part of the cellulose to the hydrated condition, for cellulose is converted into hydrated cellulose due to the mechanical action of the beater. The degree of hydration is controlled by the length of time of the beating operation and by adjustment of the beater itself. The degree of hydration controls the rate of burning of the finished powder and also reduces the flame, and I have found that nitrocellulose containing as high as 13.2% of nitrogen can be hydrated to such a point that its burning rate is no greater than that of raw cellulose. Thus by controlling the time and adjustment of the beating operation I can control the burning rate of the finished powder to any desired degree.

But I am not limited to this method of control as I have other means of control in this same operation. For instance, I can use a mixture of nitrocellulose of different degrees of nitration and so obtain a finished product of any desired degree of nitration. Then too I can add inert material such as raw cotton to the mixture in the beater. This not only slows down the rate of burning of the

finished powder but also adds materially to the mechanical strength of the finished product.

Another advantage of adding raw cotton in the beating operation is that the raw cotton can be hydrated more readily than the nitrated cellulose so that I can take advantage of both the amount of raw cotton as well as the degree of hydration to which it is brought for controlling my finished product.

As mentioned above, one of the advantages of adding raw cotton is that I can obtain a stronger sheet of nitrocellulose paper. Nitrocellulose alone when formed into a sheet of paper without hydrating any of the fibers has very little mechanical strength due to the brittleness of the fiber itself. By adding raw cotton and allowing the same to hydrate during beating, this lack of strength is entirely overcome and the fibers themselves appear less brittle, due to their being coated with a film of hydrocellulose.

Another interesting as well as important effect, traced directly to both the addition of raw cotton and to the hydration of the nitrocellulose is the elimination of static. This is especially apparent in the calendering operation where most trouble from static charges appears.

While I have mentioned raw cotton as an addition agent in the beating operation I do not wish to limit myself to this particular form of cellulose as I can obtain similar results by using cellulose in other forms such as wood pulp, flax, straw, jute, etc.

After the pulp has been beaten to the proper consistency it is transferred to what is known in the paper industry as a stuff chest, which is a large vat or tank containing a mechanical agitating device. Its function is to retain the stock as received from the beaters and to permit it to be diluted to the proper consistency for felting. At this point this process provides another important control of the properties of the finished material, for it is in the stuff chests that the various modifiers, such as deterrents or accelerators as well as stabilizers may be added as required for any particular purpose. Among the deterrents which can be added in the stuff chests are starch, either raw or gelatinized, casein, glue, nitro compounds such as dinitrotoluene, nitroguanidine, tricresylphosphate, camphor and waxes, such as paraffine, ceresine, or even such compounds as latex rubber. In adding deterrents such as dinitrotoluene, waxes etc., which have a comparatively low melting point, we take advantage of the fact that in some of the later operations sufficient heat is applied to the formed sheet to melt these bodies and thus form a homogenous coating on the individual fibers which make up the sheet. I have therefore a very efficient means as well as one which can be closely controlled for controlling the rate of the burning of the finished powder.

Powder made in this manner will require the addition of a stabilizer such as diphenylamine; this is best added to the pulp in the stuff chest, although in some instances it can be added during some of the later operations.

From the stuff chest the pulp is taken to a paper machine after passing through a series of screens to remove any particles of foreign material. Here the fiber is felted and formed into a sheet while the water is extracted and the sheet condensed to meet requirements. The paper machine may be either of the cylinder type, of the Fourdrinier type, or it may be a combination machine. Which ever type of paper making machine is used the fiber is felted and formed into a sheet in the same manner as in the manufacture of paper. The sheet of nitrocellulose as received from the wet end of a paper machine can be dried by passing it over a regular paper machine drier. Such a drier consists of a series of gear-driven cylinders heated with either exhaust or live steam. The wet sheet of nitro cellulose is passed over a series of such rolls as required to evaporate the moisture remaining after the water has been squeezed and wrung out in the paper making machine. Of course the sheet could be dried by passing it through a drying chamber.

The sheet of nitrocellulose as it comes from the driers is preferably calendered by passing between the rolls of an ordinary paper calendering machine consisting of a stand of rolls, the pressure between any pair of which can be varied to suit conditions. If necessary one or more pair of rolls can be steam heated so as to have a hot calender action. This is particularly advantageous when the pulp contains such deterrents as dinitrotoluene, waxes, etc., since the heat melts the same and secures a more uniform distribution of the deterrent. The calendering operation offers excellent opportunities for control of the burning rate of the finished powder. The greater the pressure employed in the calendering operation the greater is the density of the finished sheet; accordingly as the rate of burning varies inversely as the density the former can be accurately controlled.

The sheet of nitrocellulose paper obtained by the process above described is now ready to be made into suitable form for use as a propellant in either small arms or artillery. For use in pistols, rifles, and shotgun shells the sheet is best cut or punched into flakes of suitable size and shape. This can be done on any suitable type of cutting machine.

Before cutting into shape, the sheet of nitrocellulose can still be further modified with a resulting modification of its ballistic properties. This treatment consists in either spraying with or passing through a bath

of suitable solvent such as acetone, ethyl acetate, ether, alcohol, etc. This will colloid the surface of the sheet rendering it somewhat slower burning. As the center of the sheet is not affected by the solvent but retains its original fibrous condition this will be faster burning than the exterior colloided surface, resulting in a more progressive burning type of powder.

For large bore guns the sheet of nitrocellulose, either calendered or uncalendered, can be rolled into tubes or cylinders of any desired length, wall thickness and diameter of longitudinal perforation. The adhesive used for making the cylinder can be one that will act as a deterrent, such as glue, casein or gelatinized starch, or it can be such as material as nitrocellulose in a suitable solvent. These latter solutions can also contain gums such as kauri, copal, etc. This method of forming the sheet into cylinders, makes a very flexible process for manufacturing propellants for any size of artillery.

In accordance with the process embodying this invention, it is not only possible to produce a sheet of nitrocellulose of a single ply, but it is also possible to produce a multi-ply sheet by the employment of a multi-cylinder type paper machine, In this way, it is possibe to build up a multiple sheet of nitrocellulose; that is, a sheet made of two or more plies.

The advantage of this is at once apparent to anyone versed in the art of propellant explosives. For the sake of illustration we will consider a three ply sheet. The two outer plies can be made from low nitration cotton, say 12.6% nitrogen content while the middle or inner layer can be made of high nitration cotton of, say, 13.2% nitrogen content. I can thus build up a sheet of powder, the outer surfaces of which will have a slower rate of burning than the interior or core. This will result in a truly progressive burning type of powder. I can go even farther than this, and make, for instance, a five ply sheet containing nitrocellulose of three different degrees of nitration, progressing from slower burning in the outer layers to faster burning towards the center, or vice versa, if desired.

In addition to controlling the rate of burning of the different layers by means of the degree of nitration, I can control them by the addition of deterrents or accelerators in the stuff chest. For example, I can take a low nitration cotton, such as 12.6% nitrogen and slow it down still more by the addition of gelatinized starch or D. N. T. This will be used for the outer plies of my sheet. For the inner layer I can take some of the same nitrocellulose and increase its speed of burning by the addition of suitable compounds such as barium nitrate. As another example, I can use nitrocellulose containing raw cotton and hydrated cellulose for the outer layer pure high nitration cotton for the inner layer. This will result in a progressive burning powder, but, in addition, the high mechanical strength of the outer layer will help support the comparatively weak interior of pure high nitration cotton.

The few foregoing examples show the great possibilities of this process as worked out on the cylinder type of paper machine. They also indicate the larger number of possible combinations resulting in a close control of the properties of the finished product.

While the use of a Fourdrinier paper machine will result in a good sheet of material, it does not offer the same possibilities as the cylinder machine. On the other hand, it does not limit these possibilities but makes their application a little more awkward. It is not possible to build up a duplex or multiple sheet on an ordinary Fourdrinier machine, but even so this can be done by means of an additional operation. For example, I can make up a slow burning sheet and also a faster burning one by any of the methods outlined above. These separate sheets can then be combined into a single sheet of three plies by passing the three sheets thru a system of rolls doubling machine using a suitable adhesive. This is a common operation in paper mills and is employed for such purposes as making double lined board.

This process of forming several layers into one sheet lends itself to another valuable method of making a progressive burning powder. The two outer sheets can be made of a comparatively slow burning composition, while the inner sheet can be made from a sheet of nitrocellulose colloided with nitroglycerine. From this it is also apparent that this process can be used for making dense or colloided powders, using any solvent or colloiding agents such as acetone, nitroglycerine, ethyl acetate, ether alcohol mixture, etc.

The adhesive used can be such as a solution of gun cotton in a suitable solvent. This solution can carry such deterrents as D. N. T., etc., which are soluble in it. Gelatinized starch could also be used; this would not only decrease the rate of burning but, due to the cooling effect of the starch, would reduce the flame of the explosion, thus tending towards a flameless propellant. Adhesion could also be obtained by spraying with, or passing thru a bath of suitable solvent, such as acetone, ethyl acetate, ether alcohol, etc. This would not only act as a binding material but would also modify the rate of burning of the finished powder. Thus it can be seen that a large number of combinations with corresponding properties of the finished product can be obtained.

Having thus described the invention what is claimed is:

1. The process of making explosives comprising, converting part of the cellulose of

nitrocellulose pulp to a hydrated condition and felting or gathering the so partially converted pulp into a sheet.

2. The process of making explosives comprising, beating nitrocellulose pulp so as to cause partial hydration thereof and felting or gathering the so partially hydrated pulp into a sheet.

3. The process of making explosives comprising, beating nitro-cellulose pulp so as to cause partial hydration thereof, and continuously felting the same into a continuous web which is thereafter continuously passed through driers and calenders in order to produce the finished sheet.

4. A process of making propellant powders in which nitro-cellulose is pulped by beating, gathered from a stuff chest and felted into a sheet, characterized by the feature that the beating is selectively carried on to an extent sufficient to reduce the degree of nitration of the nitro-cellulose.

5. A process of making propellant powders in which nitro-cellulose is pulped by beating, gathered from a stuff chest and felted into a sheet, characterized by the feature that the beating is selectively carried on to an extent sufficient to partially convert the nitro-cellulose into hydro-cellulose.

In testimony whereof I affix my signature this 29th day of June, 1926.

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