

APPENDIX T

NITROCELLULOSE MANUFACTURING PROCESS

PROCESS FOR MANUFACTURING NITROCELLULOSE

The Former Gopher Ordnance Works (FGOW) manufactured nitrocelluloses – for instance guncotton (nitrogen content of 13.35-13.45%) and pyrocellulose (nitrogen content of 12.6-12.7%) – by treating either cotton or wood cellulose with a specific mixture of nitric acid and sulfuric acid.

1. Preparation of Acid Mix

1.1. Nitric acid was manufactured by vaporizing anhydrous ammonia and passing it over a platinum-rhodium gauze with a mixture of air to form nitrogen oxide (NO) and nitrogen dioxide (NO₂). When absorbed through distilled water, 60% strength nitric acid (HNO₃) was produced.

1.2 Sulfuric acid was manufactured by melting and burning raw sulfur to produce sulfur dioxide (SO₂) gas that was passed over catalytic beds to produce sulfur trioxide (SO₃) gas. It was absorbed through distilled water to produce sulfuric acid (H₂SO₄). Its purpose is to combine with water formed during reactions and prevent the dilution of the nitric acid.

1.3 The weak nitric acid was mixed with sulfuric acid to produce a concentrating mix. The nitric acid is concentrated by distillation and the sulfuric acid (that acts as a dehydrating agent) is cooled, stored and then processed to make concentrated sulfuric acid that is then mixed with the concentrated nitric acid to make a fortifying acid (about 1 part nitric acid to 3 parts sulfuric acid by weight) for the nitration process.

1.4. The waste stream from these processes was carried underground through the Laminex box sewer to the Acid Neutralization Plant. There a finely ground limestone slurry was used to neutralize the acid production waste stream before it continued its flow to the Waste Ditch along the east side of FGOW and then to the settling ponds south of the storage area. Acid neutralization was also conducted on the wastewater before it left the FGOW and flowed to the Vermillion River.

2. Preparation of Cellulose

Cotton linters (short fibers that cling to cottonseeds after the first ginning) in bales weighing 150-lbs or wood pulp in rolls weighing 700-lb were delivered to FGOW, shredded and pretreated in large ovens to reduce the moisture content to less than 1% before being blown through ducts to the nitrating building.

3. Nitration Process

3.1. About 32-pounds of cellulose fiber was mixed in stainless steel nitrators containing an approximate 1,500-pounds blend of nitric and sulfuric acids. Treated nitrocellulose (guncotton) and spent acids were then discharged from the bottom of the vats into centrifugal wringers that removed most of the acid through the outer jacket of the wringer. This spent acid was then used either for the production of pyrocellulose (pyrocotton) or fortified for reuse for the production of guncotton. Wet nitrated cotton was immediately immersed in a heavy stream of water and the slurry transferred to the Boiling Tub Houses.

3.2. Boiling - In the next step of the nitrating process, repeated washings were required to ensure uniform stability by eliminating through hydrolysis the acids and other unstable lower nitro bodies and impurities that were strongly adsorbed on the nitrocellulose fibers. In this step, numerous fresh hot water boils were conducted to reduce the acidity for a total of 70-hours before the slurry was pumped to the Pulping Houses. The water from

this step was processed through a “save-all” where nitrocellulose fibers settled out and the water recycled for boiling.

3.3. Pulping – Following the washing, pulping was conducted using machinery similar to that found in paper mills called Jordan engines. This machinery cut the fibers into very short fragments to open the fibers exposing remaining impurities in the capillary channels of the nitrocellulose fibers. A large volume of water was used. The resulting slurry was then pumped to the “poaching” house.

3.4. Poaching/Washing – The poaching step was used to reduce acidity as well as mechanically reducing the fibers to very small fragments. Additional hot water washing cycles in an alkaline medium of sodium carbonate (Na_2CO_3) was used to further eliminate acidity. Then the product was cold water washed under mechanical agitation to purify the nitrocellulose and ensure long shelf life for the powder as well as compliance with military specifications. Samples were collected and sent to the laboratory to determine the percentage of nitrogen, the solubility in the ether-alcohol mixture and the degree of fineness. It was then moved to the Blending House.

3.5. Screening/Blending – To obtain uniform propellant and ballistic characteristics, portions of batches having high nitrogen content are blended with portions of low nitrogen content. The slurry from the various poaching tubs in the previous step was fed onto mechanically vibrating screens where the nitrocellulose was blended and passed through to collecting boxes. The boxes were emptied into tubs where guncotton and pyrocellulose were blended and then sampled for nitrogen content and solubility. If satisfactory, the blended slurry was pumped to the Final Wringer House.

3.6. Wringer – The large amounts of water used throughout the process to move the nitrocellulose from step to step must be removed to ensure the colloidizing of the nitrocellulose in the ether-alcohol mix. The water was removed by the wringer with the

addition of alcohol and the semi-dry nitrocellulose was discharged into metallic, rustproof, airtight containers. Samples were analyzed to determine the moisture content remaining in the product. The containers were then transported to the Dehydration/Press House in lag cars.

3.7. The large quantities of water used throughout the nitration process (the original wastewater system designed in 1942 was to handle as much as 100,000,000 gallons of industrial wastewater daily for the six planned production lines) were sent through a system of “save-all” tanks where nitrocellulose fibers settled out, were recovered and reprocessed. Wastewater almost free of nitrocellulose particles was either returned for reuse in the poaching process or released into the waste ditch along the eastern boundary of the FGOW property.

4. Solvent Process

4.1 Dehydration/Press – After the final wringer operation in the nitration process, the nitrocellulose may still contain between 25-33% moisture. The object of the dehydration is to remove all water from the nitrocellulose and to replace it with the appropriate quantity of alcohol for a colloid with the ether added in a later step. The excess water was removed by dehydrating the slurry with a rinse of a measured quantity of alcohol forced through the nitrocellulose. Further pressure was then applied to remove excess alcohol and then the nitrocellulose formed into a block that was transported to the Mixer House on push buggies.

4.2. Mixer – The blocks of nitrocellulose were thoroughly broken up and then mixed with definite quantities of ether and a stabilizer – diphenylamine – that will absorb nitrous fumes released by the powder during long-term storage. The colloid was then moved to a macerator for further mixing and when finished, pressed into a dense cylindrical mass and moved to the Vertical Press House.

4.3. Press – The cylindrical blocks were placed into a vertical press and extruded through dies that transformed the blocks of nitrocellulose into long spaghetti-like strands. These strands fell into another blocking press that forms the colloid into the final shape. A graining press then forced the colloid through a steel die to deliver a solid strand in the form of a flexible rope. This was then cut to specific lengths depending upon the proposed use and conveyed to the Solvent Recovery Houses.

4.4. Wastewater from these processes drained through the Laminex Woodbox sewer system to the Waste Ditch.

4.5. Solvent Recovery – At this step, hot inert gases were passed through the powder to drive off residual ether-alcohol solvent gasses. The solvent vapors were condensed in a brine condenser and then pumped to a distillation building for refinement for further use. The powder was then moved by rail to the Water Dry Houses.

4.6. Hardening – The purpose of the water dry process – water was circulated around the powder until the desired volatile content was obtained – was to harden the powder and further remove moisture. Rifle powder would be then moved by rail to another facility where it was blended with dinitrotoluene (DNT) in a mixer to obtain specified burning characteristics. This step was not needed in cannon powder production. In either case, the powder was then moved to the Powder Dry Houses. Wastewater was released from the Laminex Woodbox sewer into the waste ditch along the eastern boundary of the FGOW property.

4.7. The last of the moisture was removed at the Tray Dryer and graphite added to reduce static charge and improve packing characteristics at the Glaze Barrel House. Excess graphite was removed at the Shaker and Sieve House and the powder was sampled for consistency. In order to obtain a uniform blend, the powder of different

batches was mixed at the Blending Towers after which it was packed into airtight containers and stored in one of the 96 Shipping Houses where it waited shipping instruction to move the powder to one of the ordnance plants where it would be used to manufacture shells.

Bibliography

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