The history of the manufacture of military and industrial grade Nitrocellulose

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Abstract

The manufacture of Nitrocellulose started in 1971 at the African Explosives Chemical Industry Site near Somerset West. The nitration process was a mechanical batch nitration process (according to Bonwitt). The nitration plant consisted of two batteries of four nitrators each fitted with two stirrers that rotate in the same or opposite directions. After centrifuging and washing the nitrocellulose was boiled for many hours at atmospheric conditions. For the shortening of the pre-stabilization time pressure boiling was introduced. Refining of the nitrocellulose was at first done in beaters adapted from the paper industry. At a later stage conical refiners were introduced for shortening of the fiber length. Dewatering of the NC was done in batch centrifuges.

In 1983 the manufacture of nitrocellulose started at the new NC plant at the Wellington Site. The new plant technology is based on Bowas-Induplan Chemie GmbH process technology. The Wellington plant process consists of linter bale opening and feeding, continuous nitration, continuous acid recovery, automatic pre-boiling, conical refining, automatic post boiling, blending and continuous centrifuging with the option to deliver product water wet or alcohol damp. The manufacture started in parallel with the old plant until the new plant could manufacture all NC grades manufactured by the then Somchem. The old facility was subsequently used to manufacture Carboxymethyl Cellulose (CMC) and Industrial Nitrocellulose. The old plant was shut down and demolished when the production of CMC was terminated in the 1990th.

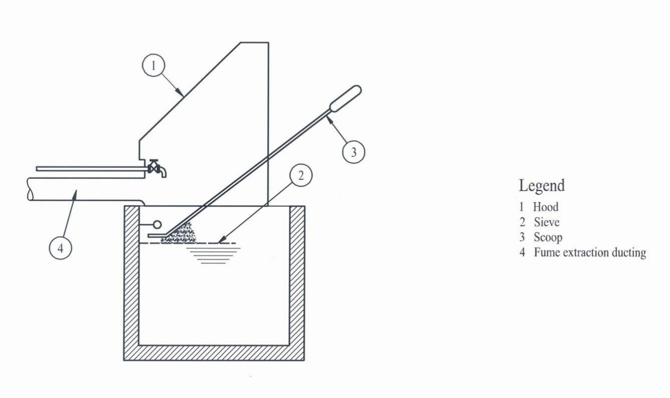
Over the last 28 years many improvements on the original Bowas-Induplan Chemie plant and process technology have been implemented. The improvements will be highlighted in the full presentation.

Industrial Nitrocellulose for the South African lacquer and printing ink industry had also been manufactured in the new NC plant, utilizing South African second cut linters, when the demand for military NC was very low. A specific grade of nitrocellulose used as an ingredient of plastic incendiary composition in the manufacture of Igniter Cord in the mining industry, was manufactured utilizing South African Softwood Pulp.

Currently Dynamite and Military grades of Nitrocellulose utilizing bulk linters are being manufactured for in-house propellant manufacture and various customers all over the world. Testing of the Nitrocellulose is done in accordance to STANAG 4178 Edition 2.

1. Introduction

Nitrocellulose manufacture started at Somerset West in the AECI factory in the early 1960th by using the displacement process.



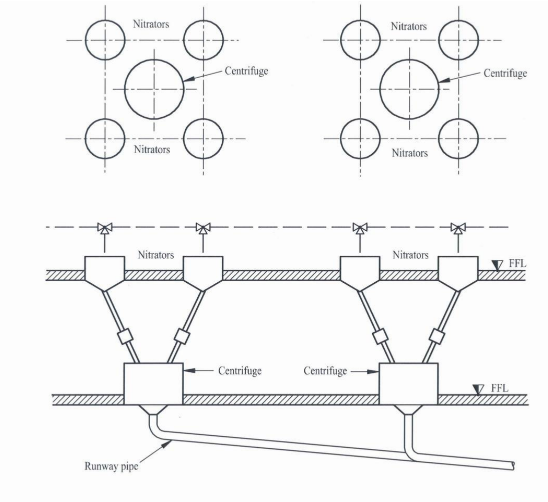
The displacement process was replaced by newer batch nitration technology by African Explosives Chemical Industry a dynamite manufacturer. This batch process is known as du Pont De Nemours process. This plant was taken over by the then Somchem (today Rheinmetall Denel Munition) in 1971. This plant manufactured nitrocellulose until 1984. A new Nitrocellulose plant was to be built at a new site.

2. **Description of nitrocellulose manufacturing processes**

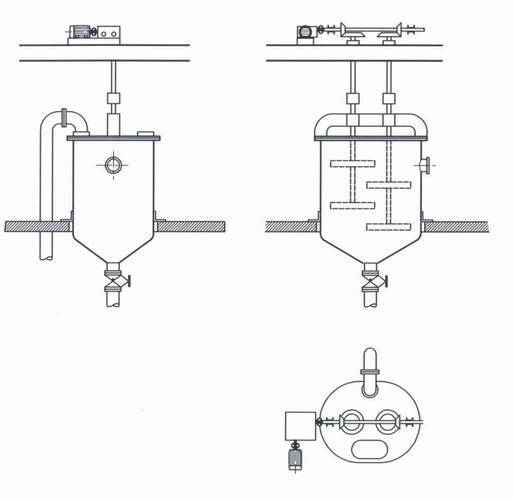
A new energetic raw material manufacturing factory was then to be built from 1980 onwards at the Wellington Site and this had to have a new NC plant, with more modern technology than at Somerset West Plant. The new process technology was bought from Bowas-Induplan Chemie GmbH and it was planned to have continuous nitration, pre-stabilization in a conicell, refining with conical refiners and post-stabilization before blending and packing. The conicell concept unfortunately did not work and was abandoned at an early stage. Instead 5 pre-stabilisation vessels were installed and the pre-stabilization process was optimized to attain Gun Cotton with excellent stability.

Whilst still manufacturing nitrocellulose at the Somerset West Factory NC plant with the old batch nitration process, commissioning of the new NC plant in the present layout at the Wellington Site started in 1983.

The Somerset West plant process consisted of two batteries of four batch nitrators fed manually with purified cotton linters (second cut and millrun) and manual acid addition, centrifuging and washing, pre-boiling manually at atmospheric conditions for a few days, (pressure boiling with a hand operated pressure boiler in later years), refining and many hours of post-boiling before blending and dewatering in batch centrifuges.

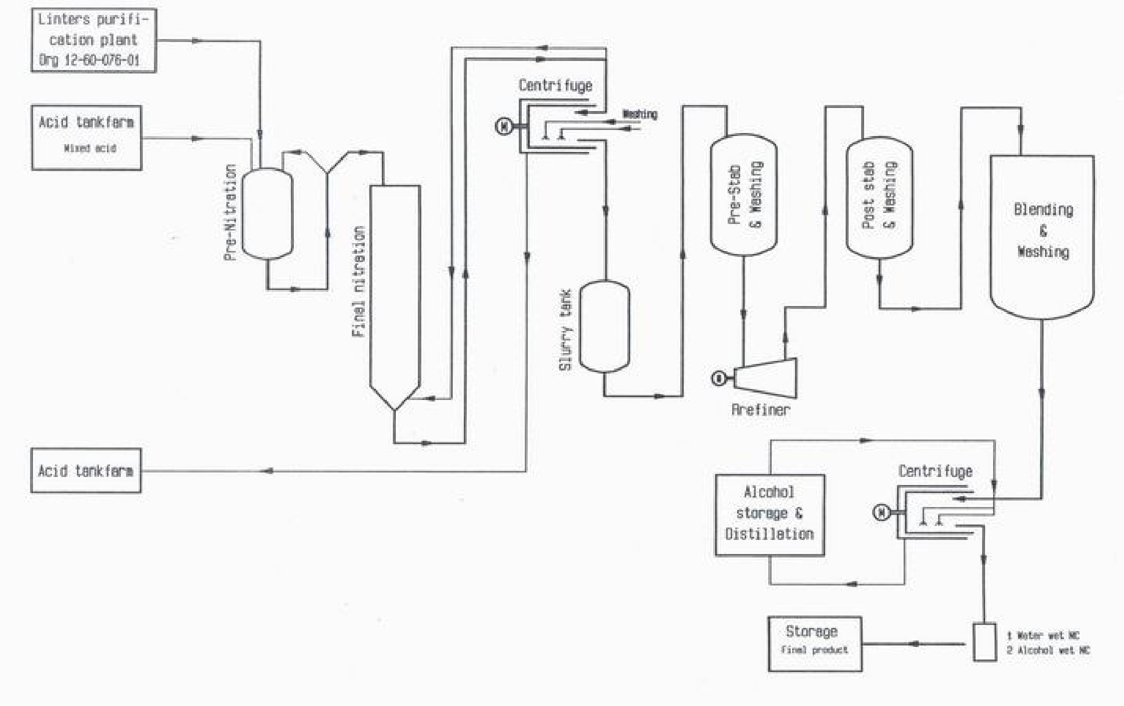


NC Nitration



NC Nitration plant

The Wellington plant process consists of bulk linter bale opening and feeding, continuous pre-nitration (meaning continuous linter and mixed acid feeding to the pre-nitrator), post-nitration, continuous acid recovery by centrifuging, automatic plc controlled pre-boiling within a 10 hours, refining, automatic post-boiling for 10 hours, blending and continuous centrifuging with the option to deliver water wet or alcohol damp NC.



Manufacture at the new site started in parallel with the old plant at Somerset West. Once the new plant was running at 90 % capacity the old plant was shut down. Part of the old plant was then modified to manufacture Carboxymethyl Cellulose (CMC) and Industrial Nitrocellulose. The CMC was sold into the platinum mining industry as flotation agent. The mining industry at a later stage switched from CMC to Guar Gum as flotation agent.

During the end of the 1980th AECI started to shut down their mining explosives manufacturing activities at the Somerset West Site in order to consolidate activities at their Modderfontein Factory in Johannesburg. The aim was to rehabilitate this land and develop it as a general light industry hub. This decision by AECI forced the then Somchem to demolish the old linter purification and nitrocellulose manufacturing plants. The demolishing was completed by 2004.

During the years where the demand for military grade nitrocellulose was low, Industrial Nitrocellulose for the South African Lacquer and Printing Ink industry had also been manufactured in the new NC plant, utilizing both softwood pulp and second cut linters.

Over the last 28 years many improvements on the original Bowas Induplan Chemie plant and process technology have been implemented. The improvements are highlighted below.

3. Continuous plant and process improvements over the last 28 years

3.1 Changes to the cotton feeding system

At first cotton was dried before nitration from a moisture content of circa 6.5 % to 1%. It became general knowledge to nitrocellulose manufacturers that too much/harsh drying of cotton fibers was detrimental to the nitration ability of linters and resulted in incomplete and un-homogeneous nitration. This was later changed by removing drier drums and using a simple conveyor system. The cotton is now fed into the nitrator with circa 6 % moisture.

3.2 Changes to the nitration acid cooling system

The Wellington area can in summer have temperatures of up to 45°C. These high temperatures necessitated changes to the acid cooling and heating system. The ability to cool acid better in summer and manufacturing dynamite type NC, called BSNC, requires nitration at very low temperatures in order to attain the required high viscosities. Before the modifications one could cool acid to between 20 and 30°C. This was done by using a freon chilling unit sending cooling water to two plate type heat exchangers to cool nitrating acid. This equipment was exspensive to maintain as it frequently needed cleaning out of NC fines and subsequently new sealing rubbers which were hard to obtain. It caused much downtime. One then designed a shell and tube set of heat exchangers where cooling is done by ammonia gas. Acid could now be cooled to 6°C for the manufacture of BSNC destined for AECI Modderfontein in the early nineties.

3.3 Improved stirrers in the acid mixing tanks at the NC plant

The plant has storage capacity of 90 cubic meter volume of acid for continuous nitration. At a rate of about 14 m^3 per hour the acid pool circulates every 6 hours — we do an analysis of the mixed acid before use and do corrective acid dosing if required. Due to the need for faster accurate results we decided to improve stirring. A Company, Atlas Starr designed stirrers which would give 30% improved hydraulic performance with the same shaft, motor and gearbox assembly. This improved our acid results and reduced the amounts of adjustments required to get the mixed acids into the required compositions.

3.4 Changes to the acid dosing system with batch and continuous dosing

The acid dosing systems were upgraded to give more accurate amounts of acid. Changes were made to the buffer vessel (acid) to stop accumulation of NC fines leading to fume offs. The original design had baffles and a stirrer on top of the buffer vessel. Over time fine NC would build up in this tank and float on top of the liquid surface once the stirrer was stopped, resulting in some major fume off's. The removal of the baffles and the stirrer solved the problem.

3.5 Improvements to the nitration process

At first a pre-nitrator and 2 final nitrators in series was used for nitration. The resulting retention time was long and stopping and starting of this system was difficult. One final nitrator column was removed from the loop and with shorter retention times and easy stopping and starting capability the product stability was much easier to achieve.

3.6 The re-design of the acid recovery system to a low maintenance system

The original system as built by the contractor would draw centrifuged acid through filters by use of a mono pump and pump the acid back to the centrifuge. These filters would block within minutes — causing much delay. The filters were removed and stainless steel lobe pumps installed — these would spray through nozzles into the centrifuge — acid not being filtered but pressure switches on the lines to stop a pump if a blockage would occur. This system worked well for 15 years and was updated using centrifugal pumps. This system is presently still in use.

The re-design of the drum filter to remove fines from spent acid before leaving the plant to the tank farm. The original filter was modified and the sieve mesh size of the drum altered.

At first we used one of the 2 centrifuges to remove acid from the NC, we had two pipe/pump systems and could use either centrifuge. We then fitted our own design acid recovery system to one centrifuge — this gave improved acid recovery. We then decided to place two centrifuges in series, first recover bulk acid form the NC and acid recovery by the acid recovery system and then to centrifuge water wetted NC again to recover more acid adding some cold scrubbing water during this centrifuging step.

The main drive to use two centrifuges in series was to improve acid recovery and reduce time before pre-boiling. The original design used one centrifuge to remove acid and do acid recovery. By changing the pipework and centrifuging the NC again from a water medium, we could reduce the washing time in the pre-boiling units before pressure boil and run at a more constant pH. Washing in the pre-stabilizing vessels is not very efficient and very time consuming.

3.7 Revised fume absorption unit.

A change to the nox absorption unit from Plinke was made. A three piston compressor system forcing off gasses through a cooled scrubbing column with cooling water generating weak nitric acid was used (20 mbar vacuum on the pipe system and 5 bar pressure in the scrubbing tower). This worked well when it was running but often required maintenance.

A change to a set of 4 columns, serviced by a scrubber fan, cooled columns with recycling recovered acid, was installed — a simpler system. This system was taken from the demolished Somerset West Site RDX production plant and cost very little to refurbish and to maintain.

3.8 Live steam into pre-stabilization vessels and its related problems

Live steam from a 10 bar boiler is reduced to 4.5 bar at the nitrocellulose plant's pre-stabilization building. The nitrocellulose plant is dependent on steam quality. During the years the boiler station water additives and how the boilers were run, had changes. At a certain stage the nitrocellulose plant manufactured very unstable product. After in-depth investigations it was discovered that the root cause of the instability problem was changes to the water chemicals including hydrazine as part of one of the additives that were added in a new boiler water treatment formulation. Removal of hydrazine containing additives, the retraining of boiler attendants, a knock out drum in the steam line and much more interaction between the boiler house and the plant, rectified the problem. We also looked at heating cycles and how to smooth peak steam consumption by scheduling.

3.9 The upgrading of refiner vessel stirrers to newer technology

One of many stability studies done by our product development group found that some NC would remain on the refiner vessel floor during refining and cause erratic fineness results and stability values. The original pitch blade impellers in the refiner vats were changed out for a far more efficient hydrofoil profile impeller having a better power to pumping ratio coefficient. The greater efficiency is achieved by the impeller having a hydrofoil profile to its blades, (rather than merely a flat plate pitched at a fixed angle of 45°), thus minimizing drag thereby providing a higher flow rate of the blade for the same installed power. The pumping rate and hydraulic performance is thus increased by 35% for the same installed power. With this improved pumping rate when nitrocellulose to be milled was stored in the refining vats for a while causing all Nitrocellulose to settle, could easily be pumped again when switching on the stirrers and no product to be refined would remain settled in the bottom corner of the vessels. Improved stirrers and new gearboxes were also fitted.

3.10 The changing of material of refiner casings, rotors and stators as well as refiner process improvements

The changing of material type of refiner casings, rotors and stators was necessary due to poor wear qualities of the original units. The original castings would erode. New stainless steel units were made and replacement rotors and stators cast.

Longer pips/strings after refining in the NC texture.
Refiner direction was not regularly changed as one should have and ran refiner teeth until they were too short — some operator and engineering discipline solved this problem.

3.11 The upgrading of pre-stab and post-stab control to touch screen (spares for Kriegler units became obsolete)

The system as installed worked well until spares became impossible to source. Whe then installed touch screen units per vessel and this works well.

3.12 The upgrading of boiling vessel cladding

From the fixed mesh/creed stone type to hanging covers of heat resistant materials — reasons: creed stone would crack after time due to continuous vibration caused by repeated boiling cycles -- This new concept aids cleaning and maintenance and reduces maintenance costs . It improves Safety—the NC would go between the vessel and the lagging and cause flashes/flames when the pre-stab temperature would increase to about 140°C— this would melt wires and instrument pipes and cause a big safety risk.

3.13 The fitment of a product detection system in decanting lines

In order to reduce product loss during decanting of water from the blending vessels, a fiber detector system would stop decanting if product wold be decanted
The operator could with the old system draw product into the decanting line if he did not adhere to settling times of NC before decanting. The new system would stop decanting if any product is detected in the waste water drawn off.

3.14 The changing of the centrifuge from hydraulic pusher speed control to a frequency converter driven unit

The unit was beyond repair and the electric_route seemed simpler. An accurate pusher speed was needed at that time when 500 tons of industrial nitrocellulose a year were manufactured, which required different operating conditions ie, pusher speed for the different industrial NC grades.

3.15 Alcohol distillation

Two old batch stills were obtained ex Somerset West Site to concentrate weak alcohol generated during alcohol damping of NC needed for single base propellant manufacture — this was a laborious process and operator dependant — one column was converted to a continuous still resulting in a much simpler user friendly distillation process.

3.16 **NC waste water** — the plant waste water would all go to a central pit, be pumped to the waste water plant and have Calcium hydroxide added to a pH of 7 before pumping the water to the evaporation ponds---this worked for many years but lime costs escalated and clogging of waste water lines due to calcification occurred – it was almost impossible to clean by chemically or by high pressure nozzles means.
The re-design of the waste water system to split acidic and pH 7 water in order to treat cheaper and more effectively — This change resulted in no calcification in the lines anymore. This is done by treating water from pH 1 to pH 3 by adding calcium carbonate granules and then adding slurried calcium hydroxide to take the pH to 7. By manipulating the factory waste water we have reversed the calcification in the lines and now have clean pipes and much reduced waste water treatment cost.

3.17 Safety improvements for easier running and improved safety

- return line on acid from nitrator to tank farm
- to cool acid further in summer and for BSNC
- machining of impellors of pumps at back of impellor — incident — pump shot out the of building
- filling of refiner cone inners with water and 6 monthly inspection — part of the refiner shot through a double wall
- removal of creed stone type lagging to lose removable panels many fires would occur during heating to 140° C. The new lagging stopped this.

4. Nitrocellulose manufacturing capabilities

Nitrocellulose is manufactured to customer demands.

Industrial nitrocellulose for the South African market, including NC for the use in the manufacture of pigmented chips to be utilized in the printing ink industry was manufactured.

We manufactured dynamite type NC for BSNC for the mining industry.

5. Customers

Alcohol wet NC for customers in United Kingdom.
Water wet NC for customers in France, Canada, Taiwan, Singapore and Brazil.
Roller compacted NC to Greece.

6. Specification and test Methods

NC is manufacture to the customer specification, but the process does not follow the old MIL Specification process as this is too costly, time consuming and cumbersome.
NC is tested to the customer's specification by utilizing the Mil Standard and/or STANAG 4178 Edition 2 test methods.
All mandatory STANAG 4178 methods have been qualified statistically.

7. Packaging and shipping

Shipping of product for export is done in carton boxes, 40 boxes (1 box containing 23 bags of 10.5 kg dry NC) per 12 m container with a total NC content of 9660 kg (dry mass).