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# THE TIGNEY STEAM EXPLOSION PROCESS

## - EXPERIENCES FROM EUROPE

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1986

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DISCLAIMER

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As a reference, the study "Review of Chemical Products Produced from the Tigney Exploded Aspenwood Process" by Arbokem Inc.

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has a complementary views, conclusions and recommendations and is available as indicated below.



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ABSTRACT

A number of research institutes and companies, mainly in Sweden and Finland have tested samples produced by the Tigney steam explosion process or have just studied the process. Their opinions about the process and results have been collected and are discussed.

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# 1 INTRODUCTION

As one part in their base for decision on how to proceed with the development of the Tigney steam explosion process. Canada-Alberta wants information on the results of the evaluation of the process and its products in Europe.

This report covers what has been done and some conclusions are drawn about the process. As some results from experiments will be available in October, they will be included in a supplement.

# 2 EUROPEAN EXPERIENCES WITH THE TIGNEY PROCESS MATERIAL

## Saltech

The Swedish company Salén Technologies AB, called Saltech, got in 1984 an option from Tigney on the process. They started to look on the potential markets for the products and to look for potential partners as they wanted a company with experience from the forest industry as a partner.

The option has formally expired and Saltech has no licence agreement, but is still interested in the process.

In the discussions about partnership with companies in the Swedish forest based industries, these have been hesitant and doubted the commercial possibilities of the process.

Saltech has found that the market for the cellulose fraction is the most interesting one, especially then for producers of regenerated cellulose. The viscose process normally has a higher demand on DP than what the Tigney process can fulfil, so still more interesting is the Neste process, which claims that a DP of 300 is sufficient. The evaluation for these purposes will be discussed later on.

The markets for the lignin is more difficult to define, much research, however, is going on especially in Sweden and Japan on the utilization of different types of lignins. One possibility is as a phenol source - there is a demand for that in Europe.

The hemicellulose fraction is the most difficult to find markets for and the same goes for the gun fibres. There is no market in Europe for substrates for edible mushrooms, but there could be a market in Southern Europe for its use as cattle feed.

In the following the opinions and evaluations from the institutes and companies who have got samples from the Tigney material are presented.

STFI, Sweden

The Swedish Forest Products Research Laboratory (STFI) has evaluated material both from aspen and poplar wood. They conclude that the exploded wood is easily extractable, yielding a pulp with quite high lignin content but with low content of hemicelluloses. The remaining fraction after extraction had a DP of 220-240. The lignin content was even after extraction with sodium hydroxide 7-12 %, and the material had a slight brown colour. They stated that the cellulose fraction had too low strength to be used as paper-making fibres, but saw it as an interesting material for production of microcrystalline cellulose.

Svenska Rayon AB, Sweden

This company is a producer of regenerated cellulose by the viscose process. They have evaluated the cellulose fraction for that purpose. This evaluation, however, is not yet finished, but results will be reported in October. The starting material had DP 200-300, which was a little lower than expected. When they then produced viscose from it, that had a very high filtration resistance, which could be interpreted that it is a mixture of high molecular weight material and low molecular weight material. If this is characteristic for the Tigney material - it would be a serious drawback for this end-use application.

Neste Oy, Finland

As was already stated in the Batelle report on the Tigney process the Neste process for the production of regenerated cellulose was the most interesting end-use application as it demanded a DP of 300. Neste is still working on the material and in October it is possible to get a summary of their evaluation.

They have had problems with the material - a certain content of particles and also the low DP (or can it also in this case be a too wide distribution of molecular weights?) They say that even if the material had DP 300, the DP was too low for good spinning properties.

Finnish Sugar Co. Ltd, Finland

This company is among other things producing xylitol (a low-calorie sweetener) from birch hemicellulose. They have tested the hemicellulose fraction as a potential xylose-source. They found, however, very low figures: 0,2 % xylose in the water solution and after a secondary hydrolysis 1,5 %. They have also a certain production of microcrystalline cellulose and saw the cellulose fraction as a potential source for that.

Bergvik Kemi AB, Sweden

This company is mainly a converter of crude tall oil to fatty acids and resin acids - raw materials for paint industry and other chemical industries. It is now trying to widen its business and was therefore interested in the lignin fraction. After having studied the process, but without having tested any material, it has now decided not to proceed with the investigations.

KTH, Sweden (Royal Institute of Technology)

Professor Bengt Rånby has done a great deal of work on grafting cellulose and other carbohydrate materials. He has also studied the possibilities of grafting the cellulose from the Tigney process. It works well and Rånby claims that the grafted material could have a potential as so called superabsorbant material for hygienic purposes.

# 3
# COMMENTS ON THE TIGNEY PROCESS

When discussing the potential use of the products from the Tigney process some aspects on the process have appeared, which could be of certain interest.

The first aspect is if the Tigney process significantly differs from the Masonite process and other steam explosion processes. Many people doubt that and say that the inventor has promised too much, that the samples delivered have not fulfilled the specifications, that the process is so difficult to control so one will never get really reproducable results. Much of this is just their personal opinions, but so far, judging from the experiments done i Scandinavia, there is no proof that the Tigney process is unique.

The other aspect is that this process - and also many other new "pulping" processes - is a "brutal" process compared with the old kraft or sulphite processes. The short process time gives an uneven attack on the fibers - the result will be that some areas are highly degraded and some are not. This seems to explain the results from Svenska Rayon with the low and high DP fractions. If this is true, so far it is only hypotheses, it can be a serious drawback for the process.

A third aspect is that a lot of work is needed in order to make the Tigney process a commercial one. Especially the extraction step have so far been done in a "laboratory way".

# 4
# OTHER WORK ON STEAM EXPLOSION PROCESSES

There has been a considerable work done on different steam explosion processes also in Europe. The most established one as we know of is in Austria where the companies Voest Alpine and Steyrmühl together with the University of Graz have run a "commercial" pilot machine for five years. They produce among other things citric acid from the glucose part and furfural from the hemicellulose fraction.

In France there are probably two producing units making different chemical products. Their forest products research laboratory in Grenoble, C.T.P., has also worked with the steam explosion process for some years.

In Sweden the University in Lund has worked with the steam explosion process as a base for ethanol production. A Swedish inventor, his company is called Processing Improvement Systems, has been working with chemical pretreatments in order to use the steam explosion process for the production of papermaking fibres. He claims that he has got good results and is now cooperating with a Swedish company in developing the process.

# 5 CONCLUSIONS

It is too early to draw any final conclusions from the work done on the Tigney process material in Europe. On this stage the following conclusions, however, could be drawn:

- The Tigney process is one among several steam explosion processes and it is not proven by the evaluations in Europe that it is significantly better than the other.

- The Tigney process is far from a commercial process - it has too many laboratory elements - so much development work is left to be done.

- There has been most interest shown for the cellulose fraction - mainly for regenerated cellulose and microcrystallic cellulose production. Then comes the lignin fraction - with no specific end-uses, and very little interest for the hemicellulose fraction.

- The "final" evaluation for regenerated cellulose will be finished in October, 86. This will probably be crucial for the interest for the Tigney process in Scandinavia.

# THE TIGNEY STEAM EXPLOSION PROCESS

# - EXPERIENCES FROM EUROPE

# SUPPLEMENT

ABSTRACT

The results and opinions from Neste Oy, Finland and Svenska Rayon AB, Sweden from their testing of samples from the Tigney steam explosion process are compiled and discussed.

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# 1 INTRODUCTION

This supplement to the main report on the evaluation of the Tigney process in Europe contains results from Neste Oy, Finland and Svenska Rayon AB, Sweden.

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NESTE OY, FINLAND

Neste has developed an alternative to the viscose process for the production of regenerated cellulose of different types. In that, cellulose, which has been pretreated to reach a DP of 350, is reacted with urea to form cellulose carbamate. This can be dissolved and the cellulose regenerated with acid, as in the common viscose process.

Neste has done extensive development work on the process. They have used commercial dissolving pulp with high alfacellulose content is a starting material but got an interest in the Tigney-process as this was supposed to produce a cellulose fraction with a suitable DP.

Neste has compared the chemical composition of a Tigney cellulose fraction with their "specification" (a high alfa pulp which has been degraded by alkali or gamma radiation).

Table 1. Chemical composition

| | TIGNEY | "SPECIFICATION" |
|---|---|---|
| DP | 230 | 350 |
| Alfa cellulose | 83.5 % | 90 % |
| Lignin (Klason) | 1.8 % | 0.1 % |
| DCM-extractives | 1.3 % | 0.2 % |
| Ash-content | 0.5 % | 0.1 % |

The starting material in this case was unbleached steam explosion cellulose from poplar.

Obviously, the high alfapulp is much more pure than the Tigney cellulose - it needs bleaching, and Neste has done some bleaching tests at the Finnish Pulp and Paper Research Institute (KCL).

Table 2. Bleaching results

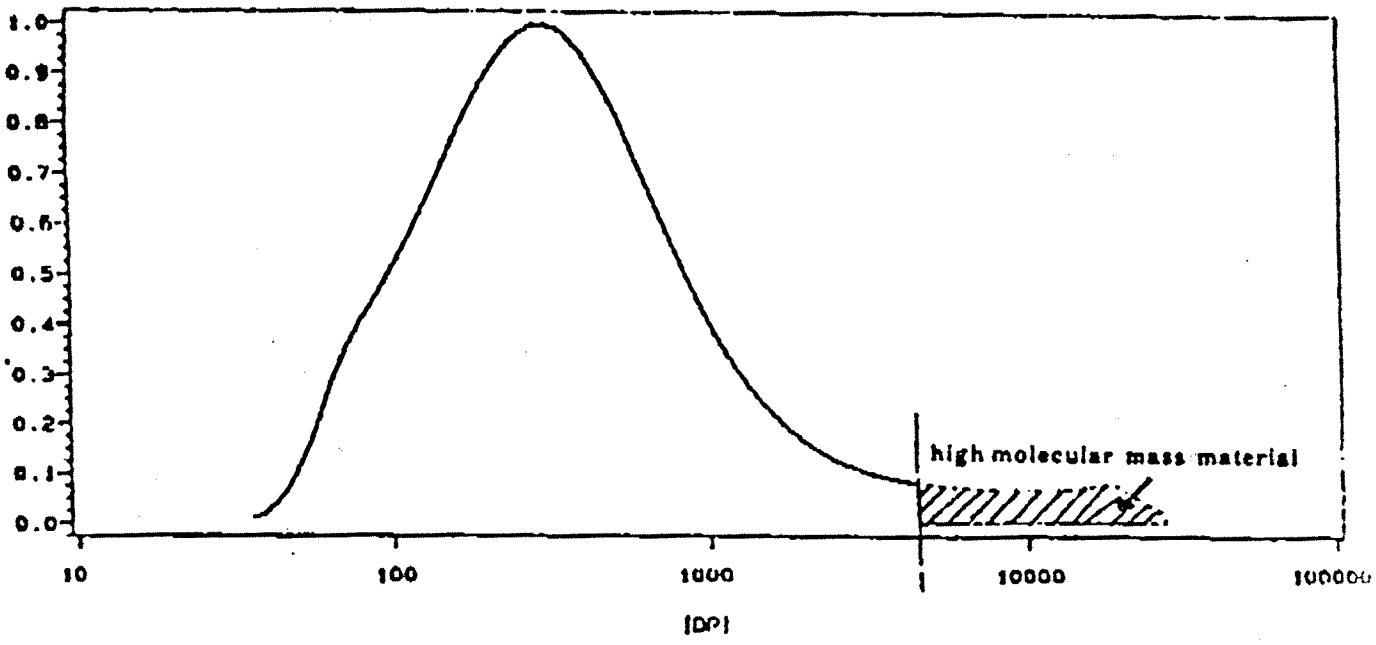
| PULP | 251/86 | | | | |
|---|---|---|---|---|---|
| BLEACHING SEQUENCE | Unbleached | | | | |
| STAGE | | D1 | E | D2 | tot |
| Consistency, % | | 10 | 10 | 10 | |
| Time, min | | 100 | 60 | 70 | |
| Temperature, °C | | 65 | 65 | 120 | |
| Final pH | | 2,6 | 10,9 | 4,5 | |
| Active Cl, % | | | | | |
| Dose/Consumption | | 2,5/2,0 | | 2,0/1,3 | 4,5/3,3 |
| NaOH dose, % | | | 1,2 | 0,2 | 1,4 |
| Yield, % | | | | | 97,4 |
| Cl-number | 1,5 | 0,8 | | | 0,33 |
| Brightness, ISO | | | | | 88,1 |
| Viscosity, SCAN | | 154 | | | 163 |
| DCM, % | | | | | 0,8 *) |
| alfacellulose, % | | | | | 81,1 |

*) Pulp sticky, some extract left on walls

Neste draws the conclusion from the bleaching test that the pulp is easy to bleach.

The problems comes when the carbamate from the Tigney cellulose is dissolved. There are highmolecular compounds in the solution causing clogging problems. Neste claims, that what they call "clogging value" is 10 times higher than it should be. The high molecular weight part is shown in Fig. 1.

FIGURE 1




NORMALIZED MOLAR MASS DISTRIBUTION

SAMPLE. No 251/86, DPw = 1114, POLYDISPERSITY = 4.81
DPw = 524 x) POLYDISPERSITY = 2.38 x)

x) high molecular mass material excluded

The DPw in this diagram is not the same DP as discussed earlier.

Neste claims, that they are still interested in the Tigney cellulose as a raw material for their process, but

- they cannot use it as it is today for production of rayon fibres - too low DP.
- it has to be bleached - too high lignin content.
- it causes processing problems - clogging.

Furthermore, Neste is directing its development resources towards the end-uses of their material, not towards raw materials for it. They know they can handle the commercial high alfa pulps - the raw material will not be the bottle neck. This means that Neste will not act as a pushing factor for the Tigney process.

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SVENSKA RAYON AB, SWEDEN

The following table shows the evaluation of some samples of the Tigney cellulose from the viscose mill's point of view.

Table 3. Comparison of Tigney cellulose and viscose pulp

| | Target value | Tigney-samples 860212 | 860606 | 861003 | Present pulp supply |
|---|---|---|---|---|---|
| R18, % | >90 | 85.7 | 84.4 | 84.2 | 93.9 |
| R10, % | >85 | 73.3 | 35.5 | 66.9 | 89.7 |
| "Rest-hemi", % | | 12.4 | 48.9 | 17.3 | 4.1 |
| Alfacellulose, % | >88 | 79.5 | 60.0 | 75.6 | 90.8 |
| Viscosity, cP | ~20 | 3.5 | 1.9 | 2.9 | 17.9 |
| Ash, % | <0.15 | - | 0.42 | - | 0.05 |
| Resin, % | <0.4 | - | 1.8 | - | 0.17 |

The second Tigney-sample, 860606, had been treated with alkali and obviously had degraded to a high extent - the first and second sample are more "representative".

R18 - the rest after extraction with 18 % NaOH gives an indication of the content of hemicellulose. The difference between R18 and R10 (after extraction with 10 % NaOH) is called "rest-hemi" and gives an indication of the amount of degraded cellulose in the sample. The Tigney samples have here much higher figures than a high alfa pulp. The amount of alfacellulose is also much lower in the Tigney samples.

The most critical property in this case is the viscosity. That gives an indication of the DP of the material. The target value is 20 cP, but the Tigney-samples have got values around 3 cP.

When processing the Tigney-samples they showed very high filtration resistance, which Svenska Rayon interpreted that the material was a mixture of high molecular weight material and low molecular weight material.

The rayon produced from the samples did not give sufficient strength properties. That because of the low DP. Svenska Rayon consider the material tested as not being suitable for rayon fibre production.

Svenska Rayon is cooperating directly with E.A DeLong and still seems to have a certain interest in the process. However, they are decreasing their production of rayon because of lack of profit and will not push the development of the Tigney process.

# 4 CONCLUSIONS

Both Neste and Svenska Rayon have come to similar conclusions about Tigney cellulose in their processes:

- impurities are causing process problems.
- low DP gives products with unacceptable quality.

Both companies claim that they still have an interest in the process. However, using a new type of raw material is not of primary interest for either company. This means that they will continue to test samples when delivered, but not take any deeper interest and not involve themselves financially.

If low DP (less than 300) and impurities (lignin, high molecular parts and low molecular fragments) are typical for Tigney cellulose more or less by the nature of the process, one must draw the conclusion that it is not suited for these end-uses.