Emerging Delignification Technology

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INTRODUCTION

My presentation is based on laboratory and mill scale pulping and bleaching experiences during the last 30 years. My involvement has especially been in the field of kraft and polysulphide pulping, in oxygen delignification and in TCF-bleaching.

The main driving force behind my research and development activities has been improvement of pulp mills profitability through wood saving, increased production capacity and cost efficient pollution abatement.

IMPROVEMENT IN KRAFT PULPING THROUGH POLYSULPHIDE AND AQ.

The main reason for applying polysulphide pulping in kraft mills is to increase the pulp yield through stabilisation of the carbohydrates in wood against alkaline degradation (peeling) (1, 2). Addition of AQ in alkaline pulping enhance both the delignification rate and increases pulp yield (3). The pulp yield increasing effect of polysulphide and AQ is additive or even synergistic (4).

The yield increase by polysulphide pulping is mainly dependent on the concentration of the polysulphide sulphur in the impregnation and cooking liquor, the impregnation and pulping conditions and wood specie (5). Reduction in chip size, improved impregnation of the chips and reduction in cooking temperature increases the yield effect. The yield gain decreases as the lignin content in the pulp diminishes. Hardwoods give somewhat lower yield gains than softwoods.

The practical mill application of polysulphide in kraftpulping is at polysulphide sulphur charges of approx. 0.5 to 1.5% based on wood. Obtainable yield increase by having certain amount of PS-sulphur present in the cooking liquor is the range of 1-2 times the amount of PS-sulphur, dependent on the impregnation and cooking techniques, kappa no. and wood specie (5). By impregnation of the chips with a Na_2S_4 - liquor followed by alkaline pulping the yield gain may be increased to 2, 5 to 4 times of the charged polysulphide sulphur (5).

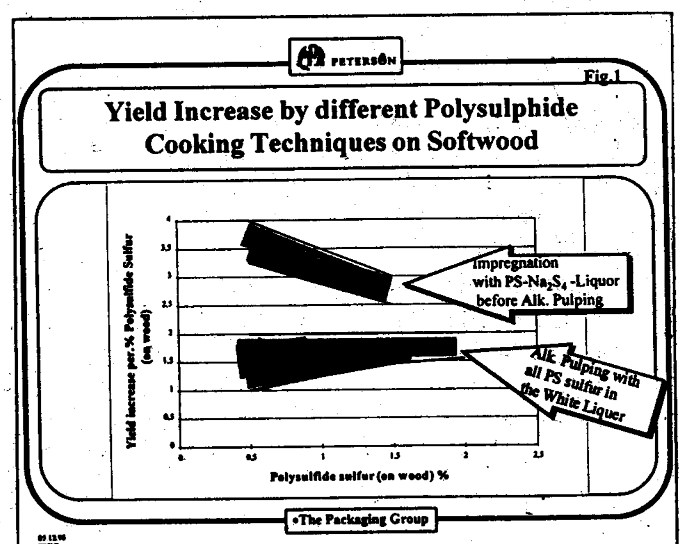
The yield gain by the different polysulphide cooking techniques is demonstrated in fig.1.

The properties of polysulphide pulps are comparable to the properties of kraftpulps. They are, however, more easily beaten than kraftpulps due to the higher content of hemicelluloses.

Addition of 0.5 to 1 kg AQ per ton of pulp in kraft and polysulphide pulping results in a yield gain of about 1% (on wood), 2-5% saving of alkali and 2-3°C reduction in the cooking temperature (4).

At present polysulphide pulping is practised in 4 kraft mills in Japan, one in Austria and the Peterson mill in Norway.

At the Peterson kraft mill in Moss we have applied polysulphide pulping since 1973.



M. Peterson & Son As,
Moss, NORWAY

The Moss mill is an integrated pulp and paper mill producing linerboard specialities from softwoods (pine and spruce). The pulp mill has a production capacity of 550 ton/day and the pulping takes place in a Kamyr dual vessel digester. The kappa no. of the pulp from the digester is at a level of 65, and the white liquor sulphidity ranges from 32 to 35%. About 0.35 kg AQ per ton of pulp is added to the cooking liquor.

The polysulphide cooking liquor is perpared by catalytic air oxidation of the sulphide in the white liquor by the Moxyprocess (6).

The flow sheet of the Moxy system, operating successfully at the Moss mill since 1976, is shown in fig.2.

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Fig.2a

Flowsheet for Moxy system

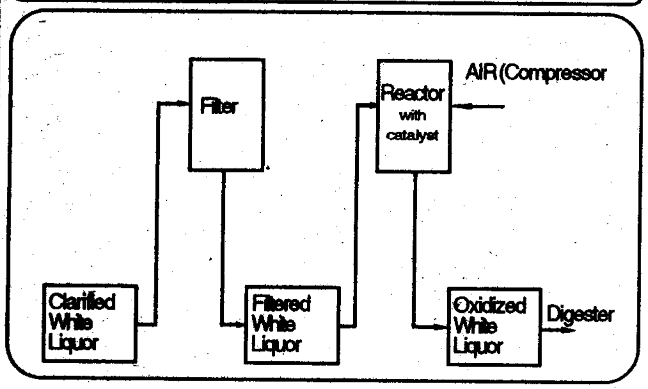

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Fig.2b

The result of polysulphide-AQ-pulping at the Moss mill can be summarised as follows:

* Reduction in wood consumption (per ton of pulp) ~ 4,3%.
* Increased pulp production capacity of the Kamyr digester ~ 4,5%.
* Increased pulping capacity when chemical recovery is a "bottleneck" ~ 10%.
* Corrosion protection of the Kamyr digester.
* Less odour formation (org. sulphur compounds) during pulping.

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The system consist of two parallel reactors containing totally 6, 5 ton of a special carbon catalyst. The throughput of white liquor is about 60 m^3/hr with only a few minutes retention time in the reactors.

By operating with a "clean" catalyst about 70% of the oxidised sulphide is converted to PS-sulphur at an oxygen/sulphide molar ratio of 0.35 - 0.4. The rest of the oxidized sulphide forms thiosulphate which is an inactive cooking chemical. The best pulping results are obtained when 50-60% of the white liquor sulphide is oxidised.

The efficiency of the Moxy system in very much dependent on the cleanliness of the white liquor and good filtration is very important. The suspended $CaCO_3$ from the white liquor absorbed on the catalyst bed have to be removed by acid cleaning, normally required every 2 - 3 weeks. More frequent washing is needed at the end of the catalyst lifetime.

The lifetime of the catalyst, one to three years is very much dependent on the suspended solid content of the white liquor feed to the Moxy reactors. The efficiency of converting sulphide to polysulphide is sometime reduced to less than 50% before the acid cleaning.

At the Moss mill the polysulphide concentration of the cooking liquor is about 5 - 6 gram sulphur per litre, which occurs when about 50% of the sulphide in the white liquor is oxidised.

The result of polysulphide-AQ-pulping at the Moss mill can be summarised as follows:

* The reduction in wood consumption (per ton of pulp) is about 4, 3%.
* The increased pulp production capacity of the Kamyr digester is about 4, 5%.
* The increased pulping capacity's when chemical recovery is a "bottleneck" is about 10%.
* Corrosion protection of the Kamyr digester is achieved.
* Less odour (org. sulphur compounds) is formed during pulping.

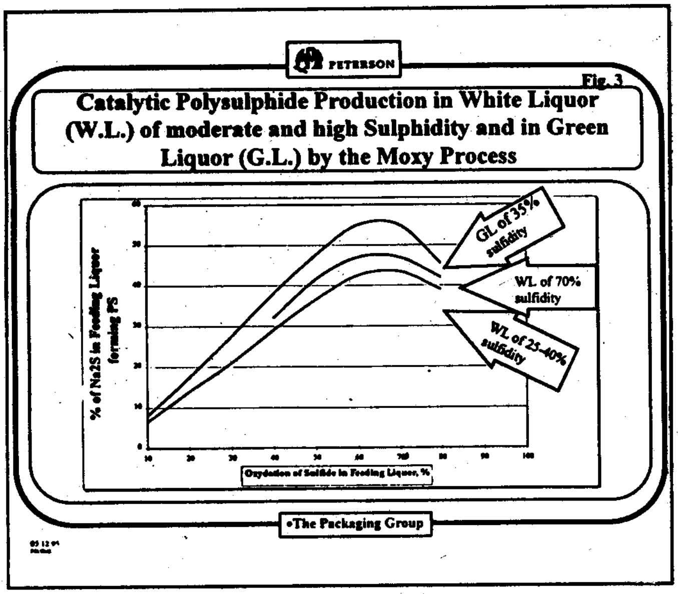
Further improvement in polysulphide pulping can be made by:

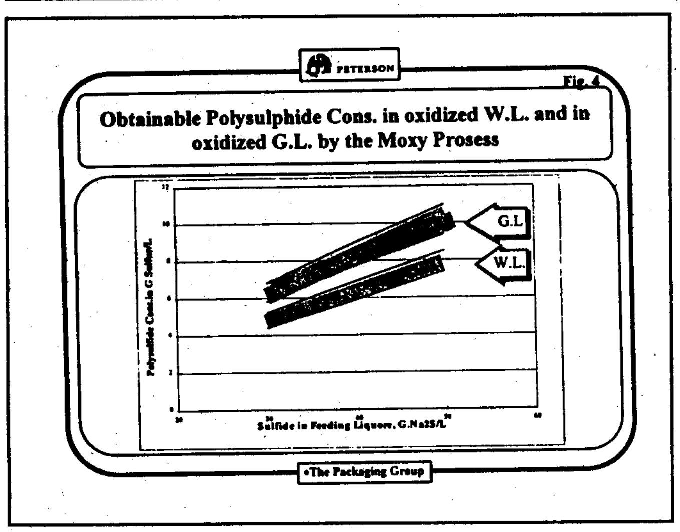
1. increasing the efficiency of converting the

sulphide present in the chemical recovery system to polysulphide, and/or

2. improving the yield increase for a given polysulphide charge to the digester system.

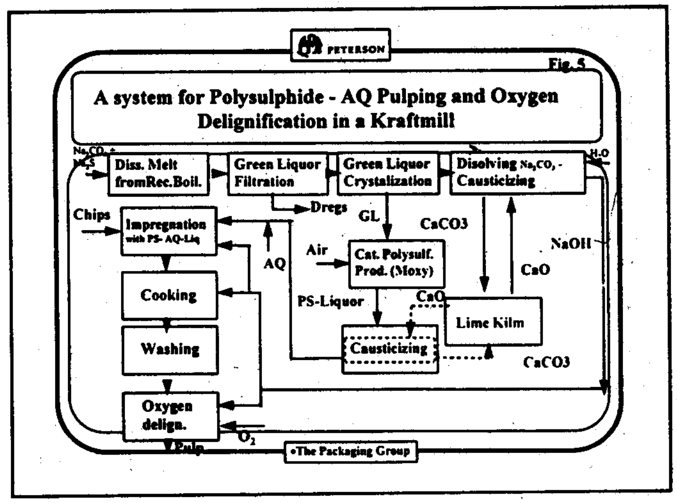
Pilot Moxy trials at the Moss mill has shown, as demonstrated in fig.3. that the efficiency of converting sulphide to polysulphide by the catalytic airoxidation process can be improved by increasing the white liquor sulphide or even more by using green liquor instead of white liquor. The improvement in





the polysulphide content of the cooking liquor by oxidising green liquor instead of white liquor is demonstrated in fig.4.

A proposal for applying polysulphide-AQ pulping



including oxygen delignification for future kraft mills is shown in fig.5. By separating the main part of the Na_2CO_3 from the Na_2S present in the recovery boiler melt, a high sulphidity liquor can be produced. The separation of the Na_2CO_3 can either be done through fractional dissolution of the alkaline melt or as here proposed by crystallisation of the Na_2CO_3 from the green liquor.

The high sulphidity liquor can selectively be oxidised through a Moxy system to Na_2S_{3-4}-liquor. This liquor should with or without caustisizing be used for impregnating the wood chips in the first pulping stage in order to give the high yield increase, demonstrated in fig.1 and improved pulp properties. The separated Na_2CO_3 from the recovery boiler melt should after dissolving in water and causticizing be used as the main alkali source for the pulping process and as NaOH for oxygen delignification.

OXYGEN DELIGNIFICATION

The application of oxygen/alkali delignification has been going on since 1970, when the first comercial installation at the SAPPI's Enstra kraftmill i South Africa was started up. The installation was based on high consisting technology. The first medium pulp consistency oxygen delignification plant was commissioned in Norway at the Peterson Moss kraft mill in 1980.

Today oxygen delignification is applied world wide in more than 100 pulp mills. The medium consistency technology is most frequent used, due to rather low capital investment cost and good process control possibilities.

The main driving force behind oxygen delignification has been reduction in water pollution due to the possibilities of replacing the elementary chlorine bleaching stage. In most cases the organic in the effluent from the bleach plant as well as the consumption of bleaching chemicals are reduced by more than 50% by oxygen/alkali delignification before bleaching. The dissolved solids from the oxygen delignification stage is returned to the chemical recovery cycle by counter-current washing.

Recently almost zero effluent pulp mills are made possible through replacing all chlorine containing bleaching chemicals by oxygen, ozone, peracids and peroxide in combination with closed counter current washing system.

The main reason for the installation of the oxygen delignification at the Moss mill was to make a cost efficient pulp production through simultainously producing two pulp grades from the Kamyr digester-a 60 Kappa no. pulp for liner board and sack paper production and a high hemicellulose containing 30 kappa no. pulp for greaseproof paper production. The low kappa no. pulp is bleached through a C (EOP) HP- sequence to 85% ISO brightness before papermaking.

During the first 10 years the delignification was made in a single oxygen reactor unit. In 1989 the oxygen delignification unit was converted into a two stage system. The purpose with the reconstruction was to prolong the delignification from kappa no.'s of 60-70 to kappa no.'s of about 22.

The present oxygen delignification plant is depicted in fig.6. It consists of the flow-splitting device (coarse screen) in the blow line of the dual vessel Kamyr digester, followed by a receiver standpipe, a MC-pump, a MC-screen, a pressure diffuser washer, a disc refiner (mixer), a $13m^3$ reactor, a MC-mixer, a $46m^3$ reactor (old), a blow tank and wash process.

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Fig. 6

Two Stage Oxygen/Alkali Delignification System at the Moss Mill

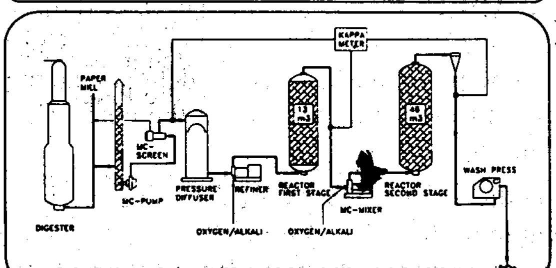

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Fig.7

A Moss Mill Trial on extended 2-Stage Oxygen Delignification of a High Kappa No. PS. Pulp

*	**Kappa No. from the digester:**	**64**
*	**Alkali Charge, as NaOH on pulp:**	**5,5%**
*	**Oxygen Charge:**	**4,0%**
*	**Reactor Temp.:**	**112°C-122°C**
*	**Kappa No. reduction:**	**42**
*	**Delignification:**	**66%**
*	**Pulp Properties: The same as for 50% delignification**	

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The production capacity of the very well functioned MC-consistency oxygen/ alkali delignification plant is about 150 tons of pulp per day.

Typical operation dates from the oxygen delignification plant is given in fig.7.

66% delignification is obtained in 60 min. by applying 5.5% NaOH and 4% O_2 (on pulp), at reaction temperature of about 110-120°C and a reactor pressure of 0.6 MPa.

Recently we have obtimized the oxygen delignification process even further. Pulps of kappa no. 60-70 can easily be reduced to kappa no. about

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Fig.8

Effect of Converting a Single Stage Oxygen/Alkali Delignification Reactor into a two-Stage Unit

		Singel Stage	Two Stage
*	**Kappa No. from The Digester**	**: ~60**	**~60**
*	**Alkali Charge, as NaOH on pulp**	**: ~4,2%**	**~4,2%**
*	**Kappa No. reduction**	**: ~30**	**~34**
*	**Delignification**	**: ~50**	**~57%**
*	**Temp.- and reaction time was unchanged**		

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18 without hurting the requested properties of pulp.

The effect of converting a single stage oxygen delignification mill into a two stage process is demonstrated in fig.8. It can be seen, that two stages enhances the oxygen delignification. This is mainly due to the better mixing of oxygen as micro bubbels into the pulp suspension by splitting of the oxygen charge into two mixers.

We have also found that sulphonation of high kappa no. pulps before oxygen delignification enhances the delignifications. This is demonstrated in fig.9.

At the Moss mill we are replacing about 50% of the NaOH needed for the oxygen delignification with fully oxidized white liquor. Fully oxidized white liquor is a cheap alkali source. However, the use of oxidized white liquor required an increased charge of effective alkali and oxygen for a given kappa no. reduction, as shown in fig.10.

It is well known (8.9) that oxygen/alkali delignification remove lignin more selectively than kraft pulping and that yield gained by polysulphide-AQ pulping is retained by oxygen delignification. The loss of yield by oxygen delignification is considerably lower than by any other applied chemical pulping methods. This is demonstrated by pulp yield data for the Moss mill, as shown in fig.11.

Consequently, the oxygen alkali delignification should be started at a kappa no. or a lignin level as high as possible. A considerable saving of pulping chemicals may also be obtained by oxygen delignification of high lignin containing pulp compared to convertional pulping methods (8). However, the oxygen delignification technology requires a fiberized lignocellulosic material for a successful result.

The yield gain by oxygen delignification of hardwood pulps of high lignin content may be greater than for softwood pulps, due to higher yieldlosses by alkaline pulping of hardwood to low kappa no. (5).

OZONE- "BLEACHING"

Ozone "bleaching" is at present practiced in one US kraft mill, in 4 or 5 Nordic kraft mills and in two European sulphite mills, The ozone

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Fig.9

Effect of pretreating Softwood Polysulphide Pulp (Kappa No. ~60) with Sulfite-liquor (30-40 kg SO_2/ton Pulp, pH: 8-9,5 before MC-Oxygen/Alkali Delignification at 110°C

	O-stage without pretreat.	O-stage with pretreat.
Alkali Charge, as NaOH, % on Pulp	: 4,2	4,2
Oxygen Consumption, %	: 3,5	3,5
Kappa Reduction, unit	: 32	40
% Delignification	: ~50%	~63%

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Fig.10

Oxidized White Liquor as Alkali Source by Oxygen/Alkali delignification of Softwood Polysulphide Pulp, Kappa No.~60 (mill scale, MC-Consisting)(2)

	O-stage with NaOH	O-stage OWL
Alkali Charge as NaOH, %	3,6	4,4 (+22%)
Oxygen Consumption, %	3,0	3.6 (+20%)
Kappa no. Reduction	29	29
% Delignification	~52%	~52%

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Fig.11

Relationship between Yield and Kappa-no for Conv. Kraft Pulping, Polysulphide Pulping and Oxygen Delignification, (Data from the Moss Mill, Kappa-no. range 30-70)

* **Conv. Kraft Pulping:** Y=0,14 Kappa-no+44,7
* **Polysulfide-Pulping:** Y=0,16 Kappa-no+45,4
* **Polysulfide-AQ Pulping:** Y=0,16 Kappa-no+45,9
* **Oxygen Delignification:** Y=0,09 Kappa-no+A_0
* **{Spruce wood, 35% W.L. sulfidity, 18% eff. alkali (NaOH), ~1% PS, 0,02% AQ (on wood)}**

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delignification is performed either at medium pulp consistentcy (~10%) or at the high pulp consistency (~25-30%). The main reason for the application of ozone for pulp bleaching is environmental and/ or cost efficiency due to replacement of chlorine and chlorine dioxide.

At Peterson we do have investigated the possibility of replacing the present used C (EOP) HP-bleaching sequence, illustrated in fig.12, by a ZOP-sequence for final bleaching of the oxygen delignified high yield polysulphide pulp.

The investigation showed, as demonstrated in fig.13 that it is possible to obtain pulp brightness and pulp properties comparable to the pulp bleached by C (EOP) HP-sequence. The new bleaching sequence required about 7-9 kg ozone charge per ton of pulp an convertion of the (EOP)-stage into a 110°C-oxygen stage.

At the Peterson Seffle sulphite mill in Sweden we do have been bleaching process since January 1994.

A CEPH-sequence was replaced by a ZEP-sequence for bleaching of an acid-Mg sulphite pulp. The layout of the new bleaching plant of 110 tons/ day pulp capacity is demonstrated in fig.14.

Good mixing of ozone into the 10% pulp suspension is extreamly important to obtain satisfactory bleaching results. Therefore, it has become nesseccary to install to MC-mixers in sequence. The time of reaction between ozone and the lignin in pulp fibres is less than 0.5 minute! The ozone generation takes place at 11 bar in a Megos generator from Schmidling with a capacity of 30,5 kg/h (fig.15).

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Fig.12

Present Bleaching Sequence For The Two Stage oxygen Delignified PS-Pulp Of Kappa No.~22

*C	– 1,6% Cl_2,	3.5% Consistency
*E_{OP}	– 85°C, 3% NaOH, 1% O_2, 0.9% H_2O_2,	10% Consistency
*H	– 70°C, 1% Cl_2,	10% Consistency
*P	– 70°C, 0,7% H_2O_2,	10% Consistency
* Final Brightness, % ISO:		~85

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Fig.13

Results of Lab. TCF Bleaching of a two Stage Oxygen delignified PS Pulp, Kappa No~20

*Z	– 20°C, 0,7-0,9% O_3	40% Consistency
*O	– 110°C, 3,3% NaOH,	10% Consistency
*P	– 80°C, 3% H_2O_2,	12% Consistency
* Final Brightness:		% ISO ~85-86
* Pulp Properties:		Comparable to pulp from the $CE_{OP}HP$-sequence.

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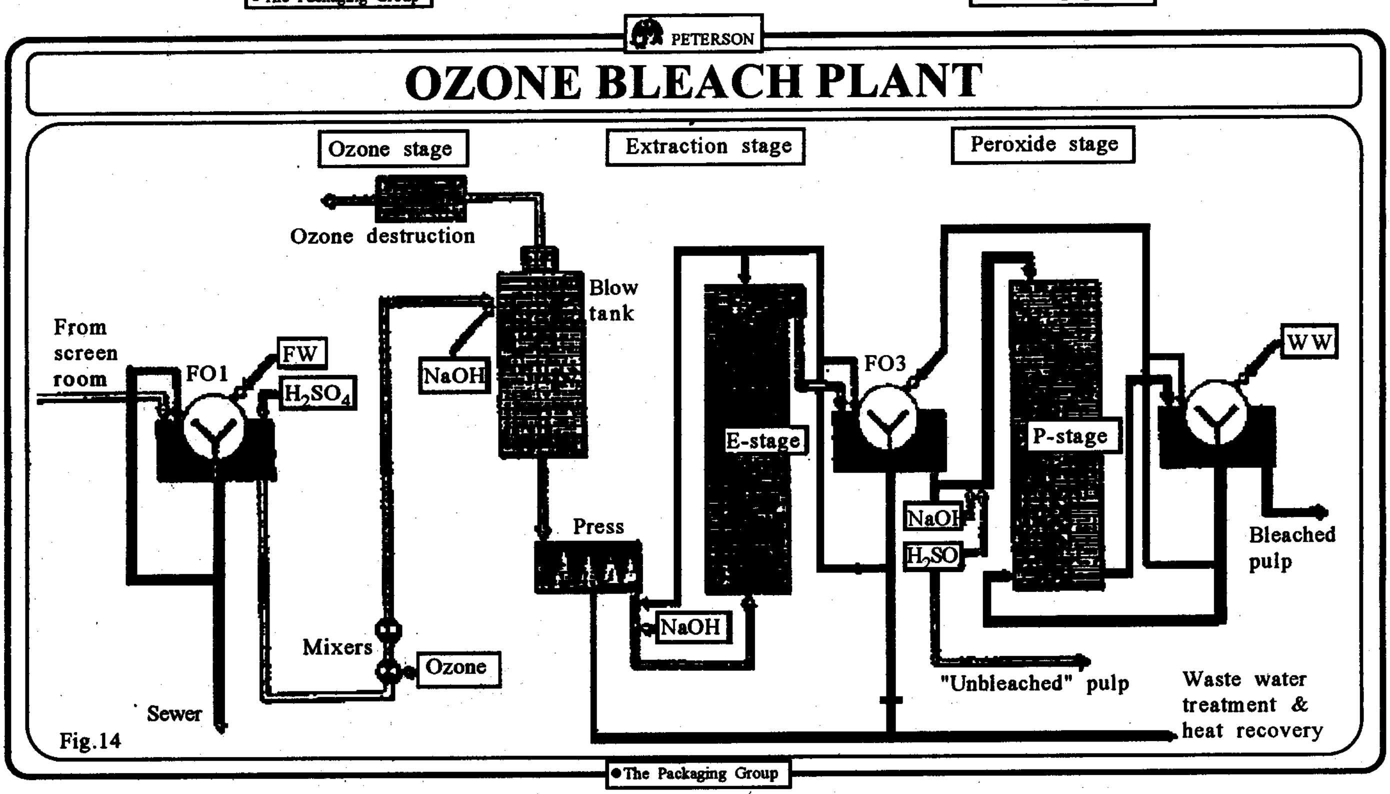

Fig.14

The charge of bleaching chemicals as well as the bleaching conditions are given in fig.16 and fig.17. The results are good, as shown in fig.18.

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Fig.15

AGA Ozone Generation Plant
Peterson Seffle AB, Sweden

- **1 Megos generator from Schmidding**
- **Ozone capacity : 30.5 kg/h**
- **Ozone concentration : 13%**
- **Gas pressure : 11 bar (a)**
- **Catalytic Ozone destructor**
- **Cooling water temp. : 2-23 degrees C**

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Fig.16

CHEMICAL CHARGES AND OPERATING PARAMETERS AT A PULP PRODUCTION OF 120 TON$_{90}$/DAY:

* Stage	:	Z	E	P
* H_2SO_4, kg/ton$_{90}$	:	1		
* O_3, kg/ton$_{90}$	:	6		
* NaOH, kg/ton$_{90}$	:		15,5	5
* H_2O_2, kg/ton$_{90}$	:			20
* Final-pH	:		10,2	10,5
* Temperature, °C	:	2-20	45	60
* Retention time, min	:	~0,5	30	360

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Fig.17

OPERATING CONDITIONS IN THE OZONE STAGE

- **Kappa No. of acid Mg. Sulphite pulp : 22-26**
- **Pulp consistency : 10%**
- **Ozone concentration in O_3 : 13 wt.%**
- **Ozone charge kg/ton$_{90}$: 6**
- **H_2SO_4 charge kg/ton$_{90}$: 1**

Kappa no. reduction : ≥ 50%
Kappa no./kgO_3/ton$_{90}$: ca.2
Ozone consumption : 99%

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Fig.18

RESULTS OF ZEP-BLEACHING COMPARED TO OLD BLEACHING SEQUENCE, C-Ep-H

- **Brightness : Unchanged, 84+%ISO**
- **Pulp beatability : Unchanged or better**
- **Resin content of the pulp : Reduced by 50%**
- **Runability of the paper machine : Unchanged or improved**
- **Paper strength : Unchanged**
- **Greaseproof properties : Unchanged**

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A pulp brightness of 84% ISO is easily obtainable by applying 6 kg ozone, 16 kg NaOH and 20 kg H_2O_2 per ton on pulp of 22-26 kappa no. The papermaking properties of the new pulp are, when compared with the old CEPH bleached pulp, equal or superior.

The ozone bleaching process applied by Peterson Seffle was forced on the mill by the Swedish Pollution Abatement Autorities, requiring a TCF bleaching process. However, the installation of the new process has been very successfull and gives a good return on the investment.

APPLICATIONS

The use of polysulphide, AQ as well as oxygen in alkaline delignification processes may increase pulp yield considerably. The yield increasing effects are additive and in the case of polysulphide/ AQ even synergetic.

A combination of PS-AQ pulping with oxygen delignification will in most kraft mills result in profitable woodsaving and sizeable improvement in production capacity. An example is illustrated in fig.19. For production of linerboard polysulphide-AQ pulping may be interrupted in the kappa no. range of 60-100 for making of the base liner pulp. Part of the base liner pulp may then be further delignified by oxygen and alkali to kappa no. 20-50, dependent on the required pulp cleanness and brightness for the top liner pulp.

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Fig.19

Application Polysulphide-AQ Pulping and Oxygen Delignification in Future Kraftmills

LINERBOARD PULPS:

- **Poly-sulphide (AQ)-Pulping to Kappa-no 60-100 for making of Base Liner Pulp followed by Oxygen Delignification to Kappa-no.~25-50 for making of Top Liner Pulp.**
- **RESULTS** — **Considerable increase in Pulp Yield and Production Capacity compared to conventional Kraftpulping.**

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Fig.20

Application of Polysulphide-AQ Pulping and Oxygen Delignification in Future Kraftmills

BLEACHED SOFTWOOD PULP:

* **Poly-sulphide (AQ)-to Kappa-no~25-50, followed by Multistage Oxygen Delignification to Kappa-no 8-12 and final TCF-bleaching: (O)OQ$_{O(P)}$ZE$_O$P or (O)OQO(P)PaaE$_O$P**

* **RESULTS** 1) **Wood Saving due to higher Pulpyield**
2) **Increased Production Capacity**
3) **Easy beaten pulp**
4) **Easy to apply a "Closed" Mill Consept and obtain a Minimum Environmental Impact Mill**

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The result of this approach could be 5-10% reduction in wood consumption per ton of pulp and 5-20% increase in production capacity compared with conventional pulping practice.

Another example is illustrated in fig.20, for production of bleached pulp grades.

Polysulphide-AQ-pulping can be interrupted in the kappa no. range of 25 to 50 and followed by multistage oxygen delignification to kappa no. 8-12 before final bleaching. The final bleaching may be a TCF-sequence.

Q-treatment before the final oxygen stage should remove harmful metals and even "activate" the lignin for improved delignification (10). According to my knowlegde will the final bleaching through a ZEoP or PaaEoP-sequence be rather cost efficient.

The combination of terminating the pulping process at relatively high kappa no. before multistage oxygen delignification and final bleaching could result in as much as 10% lower wood consumption (per ton of pulp) and 10-20% increased production capacity compared with conventional practice.

The combination of pulping and multistage oxygen delignification will also make it relatively easy to adapt a "closed" mill concept and obtain a minimum environmental impact pulp mill.

It is to assume that oxygen/alkali delignification applied in general to high lignin containing cellulosic materials will result in a considerable saving of raw materials and pulping chemicals and improved production capacity when compared to conventional pulping practices.

CONCLUSIONS

1. Polysulphide pulping has been practiced at the Peterson kraft mill in Norway for more than 20 years. The results have considerable reduction in wood consumption, improved production capacity, reduces odour formation and prevention of digester corrosion.

2. Polysulphide pulping is made possible through a catalytic airoxidation of the sulphide present in white liquor through the Moxy process. However, airoxidation of green liquor looks even more promising due to a higher efficient convertion of sulphide to polysulphide sulphur.

3. Addition of small amounts of AQ improves the yield gain and rate of delignification by polysulphide pulping.

4. Medium consistency oxygen/ alkali delignification as applied at the Peterson kraft mill since 1980, removes lignin much more selectively than any conventional pulping process.

   The two stage oxygen process may remove more than 65% of the lignin in high kappa no. Polysulphide-AQ pulp without hurting the requested properties of the pulp.

5. Ozone can replace chlorine by bleaching of both kraft pulp and sulphite pulp without hurting the required pulp properties.

6. At the Peterson Seffle sulphite mill in Sweden, has a ZEP bleaching sequense successfully replaced as CEPH-sequence since one and a half year. The medium consistency ozone delignification plant is working very good due to extremely good mixing and high ozone concentration (13%) in the treatment gas (O_2)

7. The technology for polysulphide-AQ pulping, multistage oxygen delignification and ozone bleaching successfully applied at the Peterson's pulp mills ought to have great potentials as good investments in a variety of pulp producing mills around the world.

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Asian Development Bank Initiates Energy and Environmental Audits

Asian Development Bank, (ADB), Manila apart from investing in the power and infrastructure sectors is also involved in the improvement of energy & environmental efficiency in the industrial sector in india.

ADB entered into an agreement with the Government of India in early 1995 for providing technical assistance for the "Energy Efficiency Support Project".

During July 1995, ADB invited five international consulting firms from various parts of the world to submit proposals for carrying out energy and environmental audits in India.

After rigorous evaluation, ADB selected for the project "Agency for Environment and Energy Management (ADEME) of France together with their Indian Associates M K Raju Consultants Private Limited (MKRC) of India.

The project covers 45 manufacturing plants in the six industrial subsectors-namely, Cement, Fertilizers, Mini-steel, Pulp & Paper, Sugar and Textiles. The project includes energy & environmental audits; guidelines for feasibility & pre-investment studies; training & development activities; promotion & awareness schemes.

The executing agency of the project is the Industrial Credit and Investment Corporation of India (ICICI).

The project has a three phased program:

First, identification of the energy savings potential in all the plants in all the sectors which will amount to about Rs 150 crores annually.

Secondly, a detailed study in one unit in each of the six sub-sectors with a technology audit. This serves as a model for other industries.

Thirdly, a demonstration project to highlight the new and effective technologies. This would facilitate the individual units to take up appropriate action for better energy & environmental efficiency.

ADEME, with its headquarters in Paris, France,is a state Industrial and Commercial Body with 400 technical specialists acting under the joint responsibility of the French Ministries of Environment, Industry and Research and is the essential tool for implementing French environment and energy policies.

MKRC are pioneers in energy conservation having covered over 400 companies in India, UK and South East Asia.

A project office is set up at MKRC in Madras with all necessary backup facilities including energy and environment audit instruments.

M.K. Raju, Chairman, MKRC, said "the ADB Project will give the much needed fillip to make the Indian industry globally competitive with the energy consumption standards comparable to the best in the world".

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