

WATER RESEARCH COMMISSION PROJECT NO. 122

**WATER MANAGEMENT AND EFFLUENT TREATMENT IN THE
TEXTILE INDUSTRY : SCOURING AND BLEACHING EFFLUENTS**

FINAL REPORT

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EXECUTIVE SUMMARY

The most serious water quality problem facing the Republic of South Africa is the increasing salinity of the country's water resources, particularly as the Republic is a water deficient country. Water at a quality suitable for various urban and industrial uses is the critical factor for continued economic and industrial growth.

Because of the water shortage, the return of industrial waste waters to the environment is necessary. It constitutes a considerable supplementary source of water yet adds significantly to the mineralisation process and deteriorating water quality.

The control of industrial discharges, and the encouragement of water recycling and reclamation by industry, form part of the Department of Water Affairs' strategy and policy towards the use of water for industrial purposes. At the same time, the development of processes for the treatment and recovery of effluents which contain high levels of dissolved organics and inorganic impurities, has been initiated.

Effluents from the textile industry, particularly those arising from scouring and bleaching processes, give rise to particular concern as they are in general non-biodegradable and contain high concentrations of mineral salts.

The Pollution Research Group was contracted by the Water Research Commission to investigate various process options which would enable the recycle and reuse of water, chemicals and/or, heat energy from textile scouring and bleaching effluents.

The compositions, volumes and pollution loads of scouring and bleaching effluents from the processing of wool (carbonising and bleaching only), cotton, polyester and their blends were examined.

Strong caustic effluents produced during the scouring of cotton and cotton blends were identified as the most problematic of the effluents examined in terms of pollutant type and loading. Investigations, therefore, concentrated on the development of technologies which would alleviate discharge problems encountered with this class of textile scouring effluent.

In addition, laboratory and pilot scale investigations were conducted on the :-

- (i) hyperfiltration of wool carbonising effluents.
- (ii) low temperature conventional ultrafiltration of weak polyester and polyester cotton scour effluents.
- (iii) evaporation of strong cotton and polyester/cotton scour effluents.
- (iv) electro-oxidation of strong cotton and polyester cotton scour effluents.

The most effective solution to the problem of strong caustic scour effluents was found to be a four stage treatment process.

The process sequence involves :-

- (i) neutralisation by an acidic gas,

- (ii) cross-flow microfiltration to remove suspended, colloidal and waxy contaminants from the neutralised effluent,
- (iii) nanofiltration to separate and recover the neutral sodium (or potassium) salt from the soluble organic and divalent metal contaminants,
- (iv) electrochemical treatment in a membrane cell to split the sodium or potassium salt to form hydroxide, an acidic gas and a depleted salt stream.

This treatment process was tested on a laboratory scale and pilot plant trials were conducted at three textile factories. The Water Research Commission has been granted a patent for the process in the Republic and overseas.

The acidic gas used in the neutralisation stage may be either chlorine or carbon dioxide. At Smith and Nephew, Pinetown, where cotton is kiered as opposed to scoured, the chlorine system was used. On average the pretreatment sequence of neutralisation, cross-flow microfiltration and nanofiltration removed all the colour, 60 % of the organics and the chemical oxygen demand, 85 % of the calcium and 25 % of the magnesium. Potassium hydroxide was recovered in the electrochemical unit at 90 % current efficiency.

The chlorine system was also used at David Whitehead & Sons to recover sodium hydroxide from scour effluent. Electrolysis of the nanofiltrate, produced sodium hydroxide at above 80 % current efficiency.

In both trials operational current density was strongly dependent on anolyte concentration, dropping sharply as anolyte sodium concentration decreased below 10 g/l. This effectively made it uneconomical to remove more than 55 % of the sodium or potassium from the nanofiltrate.

The carbon dioxide system was used at Da Gama Textiles to recover sodium hydroxide from scour liquor. The electrochemical stage of the pilot-plant trials recovered sodium hydroxide at an average current efficiency of 62 %. The recovered caustic stream was extremely pure and the depleted brine stream was of suitable quality for reuse in the scour wash process without pH adjustment. To overcome the drop in current density with decreasing anolyte sodium concentration, a background concentration closed loop recycle wash system was incorporated. Results showed that the operation of such a loop containing 10 to 30 g/l sodium would decrease the required electromembrane area significantly.

Some problems were encountered with the operation of the electrochemical cell which resulted in a high power consumption. Polarisation with subsequent loss of current efficiency was shown to have occurred.

Although the efficiency of caustic production was higher and power consumption was lower, when the chlorine system was used, the carbon dioxide system is preferred and the Pollution Research Group are confident that if the electrolyte flow distribution in the carbon dioxide system is optimised, then current efficiencies will be increased and power consumption decreased.

During the course of the project, effluent, water and chemical surveys were conducted at various textile factories. The subsequent recommendations have enabled significant savings in chemicals, water and pollution loads to be achieved.

A Guide describing the treatment of bleach and scour effluents in the textile industry, including design data, was prepared. A total of 6 publications, 9 conference papers and patent applications in 6 countries have resulted because of this project.

The Water Research Commission has entered into a sub-project with a soft drink manufacturer to apply the technology to the treatment of bottle wash water.

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The efforts and contributions of the many people in the Textile Industry, the Pollution Research Group, the Department of Chemical Engineering and the Water Research Commission who were involved in the project are gratefully acknowledged.

Special mention must be made of Da Gama Textiles (Zwelitsha), Smith and Nephew Ltd. (Pinetown), Ninian & Lester (Pty) Ltd. (Pinetown), OTH Beier (Pty) Ltd., Mym Textiles, David Whitehead & Sons (SA) (Pty) Ltd. (Tongaat), Consolidated Textiles, Swazi Textiles and John Grant.

These research and development investigations were guided by a steering committee for the project which comprised, over the period of the project, the following members :

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INTRODUCTION

Southern Africa is, for the most part, a semi-arid, water-deficient region of the world, subject to variable rainfall, droughts, floods and high evaporation losses. Annual rainfall amounts to only 58 % of the world average, run-off is distributed unfavourably, the availability of underground water is limited and the quality of the water resources is deteriorating.

The increasing salinity of the Republic of South Africa's water resources is the biggest water quality problem facing the country today and industry's future is largely dependent on the control of this problem. Added to this concern is the fact that, since Southern Africa is a relatively arid and water deficient region, the industrial effluent return flow forms a considerable supplementary source of water, adding significantly to the mineralisation process.

With the future need to safeguard the quality of fresh water supplies and to reduce fresh water use by the introduction of reclamation schemes, the control of industrial discharges will become of great importance. In many instances the waste that industry discharges is detrimental to the production of a high quality reclaimed water.

Recent amendments to the Water Act have placed considerable responsibility on industries to optimise their water use and to treat their effluents to required standards. The textile industry, because of the nature of its effluents, is faced with a particularly serious set of problems.

Textile processing plants utilise a wide variety of dyes and other chemicals such as acids, bases, salts, detergents, wetting agents, sizes and finishes. Many of these are not retained in the final product and are discharged in the effluent. Textile effluents are in general relatively non-biodegradable and hence present problems in terms of discharge to both sewage systems and to the environment. Mills discharging to sewage works cause colour and chemical oxygen demand (COD) problems and those discharging to the environment need to remove very high percentages of colour, COD and mineral salts.

Typical textile processing water use is 150 to 400 l/kg of product and hence large volumes of textile effluents need to be disposed of in the Republic.

In 1983 the Pollution Research Group was awarded a three year contract by the Water Research Commission to investigate water and effluent management in the textile industry with special reference to bleach and scour effluents. An extension, for one year, was granted in 1986 and for a further six months in 1987. During this period a treatment sequence was developed for the recovery and recycle of chemicals, heat energy and water from cotton scour effluents. The treatment sequence involves :-

- (i) neutralisation using an acidic gas,
- (ii) cross-flow microfiltration to remove suspended, colloidal and waxy contaminants from the neutralised effluent,
- (iii) nanofiltration to separate and recovery the neutral sodium (or potassium) salt from the soluble organic and divalent metal contaminants.

- (iv) electrochemical treatment in a membrane cell to split the sodium or potassium salt solution to form hydroxide, an acidic gas and a depleted salt stream.

The treatment sequence was tested from the laboratory scale through to pilot plant scale, and sufficient experimental data obtained to enable the design of a full scale effluent treatment plant. A patent, number 87/4406, has been granted and assigned to the Water Research Commission. Applications have also been made in a number of overseas countries.

A Technical Guide describing the treatment of bleach and scour effluents in the textile industry, including design data, was prepared.

In addition a detailed analysis of the scouring wash range at Da Gama Textiles was conducted as well as several water and chemical management surveys at various textile plants.

This final report of the project, summarises the work conducted in the period from 1983 to 1987. Full experimental details and analytical results together with theoretical aspects relating to the devised treatment sequence are given in Appendices 1 to 9.

2 TEXTILE EFFLUENTS AND WATER MANAGEMENT

The successful implementation of any effluent treatment plant is dependent on the acceptance by management that effluent treatment is only part of the overall water and chemical flow regime within a factory. An understanding of the overall water consumption of the factory, the process water distribution and the water consumption in each section of the plant is of equal importance. This knowledge allows in-house control measures to be implemented and can lead to considerable savings in terms of water and chemical usage and reduced effluent discharge. Surveys were conducted at a number of textile plants to detail the water and chemical reticulation system within the factory.

Trials have shown that substantial savings in water, effluent discharge costs and energy costs can result from the cascading of effluent from "clean" processes as wash water in processes which do not require high quality water.

A matrix of the possible cascade operation is given in Table 1.

Table 2 summarises the results of the cascading trials conducted.

Estimated heat, water and effluent discharge savings for the reuse of effluent from the second stage merceriser wash range at the factory were approximately R14 000 per annum.

An advantage of using recycled water is that the flow rate to the machines is not dependent on mains water pressure. This results in more consistent cloth quality.

TABLE 1
POSSIBLE CASCADE OPERATION

Source	Cascaded to:							
effluent	Ox desize	Scour	Bleach	Merc 1	Merc 2	Relax 1	Relax 2	Treatment
Desize								X
Scour								X
Bleach	X	X			X			X
Mercerise 1								X
Mercerise 2	X	X	X	X		X		
Relaxer 1		X						X
Relaxer 2	X	X	X		X	X	X	
Ox. Desize								X

TABLE 2
RESULTS OF CASCADE TRIALS

Source effluent	Destination process	Comment
1. Merceriser 2 (weak NaOH)	Scour wash range	1. Residual NaOH on cloth increased from 20 g/l to 40 g/l. 2. Residual TOC increased by 25 %. 3. No difficulties experienced in further processing of cloth.
2. Merceriser 2	Ox. desized wash range	1. Residual NaOH on cloth lower than when washed with fresh water. 2. Residual TOC increased by 25 %. 3. No difficulties experienced in further processing of cloth.
3. Merceriser 2	Merceriser 1	1. No increase in residual NaOH on cloth. 2. Residual TOC and TDS increased by 50 %. 3. No difficulties experienced in further processing of cloth.
4. Relaxer 2 (weak sodium acetate)	Relaxer 2	1. Residual NaOH on cloth easier to control and less acetic acid required for neutralisation. 2. No deterioration in cloth quality for reasonably short runs (5 to 6 hours).

3 WOOL CARBONISING AND BLEACH EFFLUENT

Carbonisation is the chemical destruction of vegetable matter present as a fleece contaminant. Generally the greasy wool containing vegetable matter is scoured to remove grease, grit and suint salts then passed through a standing bath of dilute sulphuric acid (5 to 7 % m/m) where the acid is preferentially absorbed by cellulosic vegetable material. The wool is then squeezed, dried conventionally and baked at a high temperature (100 °C). The vegetable matter is carbonised by the acid and becomes black and brittle. Beating and dusting removes the ash and burnt organics. The wool is then neutralised by washing in a sodium carbonate solution before further processing.

Laboratory scale tests were conducted using conventional reverse osmosis (Appendix 2). The product water was of extremely high quality with rejections ranging between 95 and 100 %. Permeate flux decline, could be effectively controlled by either the addition of an antiscalant or sequestering agent or by careful pH control at acid levels. Disposal of the low volume (5 to 10 % of initial volume) mainly sodium sulphate concentrate would be required as chemical recovery would not be feasible. Recommendations to optimise water usage at the factory were made.

At the OTH Beier plant, wool is bleached in a chloride solution containing a non-ionic wetting agent at 12 to 18 °C. The bleaching solution (chlorine water) is dumped continuously at a rate of approximately 60 m³/day. The chlorine water is a highly oxidising dilute hydrochloric acid solution contaminated with 1 to 2 g/l organic matter.

Laboratory investigations indicated that total recycle of both water and oxidising chemicals is technically feasible for this type of effluent. The proposed scheme involves the process sequence reverse osmosis, nanofiltration and electrolytic salt splitting in a manner similar to that used for sodium hydroxide recovery from strong scour effluents (Section 5.4).

4 THE TREATMENT OF WEAK SCOUR EFFLUENT

Polyester and polyester/cotton knit fabrics are scoured in weak solutions of sodium carbonate or sodium hydroxide and detergent to remove knitting, spinning and antistatic oils which are absorbed by the fibre during the various extruding, spinning and knitting operations.

Although the scouring solutions are relative weak, a treatment process which could recover the scouring chemicals from the cloth contaminants would be desirable. Preliminary investigations using various conventional flat-sheet ultrafiltration membranes against polyester scour effluent from a local textile mill showed that the Abcor HFK 132 membrane type was the most suitable membrane available for this application.

Long-term ultrafiltration trials using a 3,3 m² spiral wrap Abcor HFK 132 membrane were subsequently conducted against polyester/cotton scour effluent. The permeate recovered was crystal clear but foamed on shaking. Approximately 30 to 50 % of the organics in the effluent and 70 to 80 % of the inorganic scouring chemicals in the effluent passed through the membrane into the permeate. Despite good permeate

quality, fouling of the membrane surface occurred and fluxes dropped to 20 $\text{L}/\text{m}^2\text{hr}$ at 90 % permeate recovery. Fluxes were restored after chemical cleaning of the membrane. Appendix 3 contains detailed results of the laboratory and semi-technical scale investigations.

5 THE TREATMENT OF STRONG SCOUR EFFLUENT

Strong scour effluents are, conventionally, discharged to the environment without reuse or recovery. This has a large impact on the environment as a medium sized textile mill will discharge between 0,8 and 1,2 tons/day of sodium hydroxide into the aqueous environment in the form of scour effluent containing up to 20 g/L sodium hydroxide and contaminated with organic material.

Strong scour effluent contributes up to 25 % of both the volume and the COD loading of the total factory discharge. It is thus identified as posing a serious pollution problem.

Three processes were tested for the complete treatment of caustic effluents from the scouring of cotton fabrics and are illustrated schematically in Figure 1a, 1b, and 1c.

In the first process (Figure 1a) the effluent is treated by evaporation and centrifugation. Both the condensate and concentrate may be recycled.

In the second process (Figure 1b) the organic material in neutralised scour effluent undergoes electro-oxidation by electrolytically produced hypochlorous acid. Sodium hydroxide is recovered from the effluent in a cation-exchange membrane electrolysis cell.

In the third process (Figure 1c) organic material and divalent inorganic ions are removed from neutralised scour effluent by nanofiltration followed by the recovery of the sodium hydroxide in a cation-exchange membrane electrolysis cell.

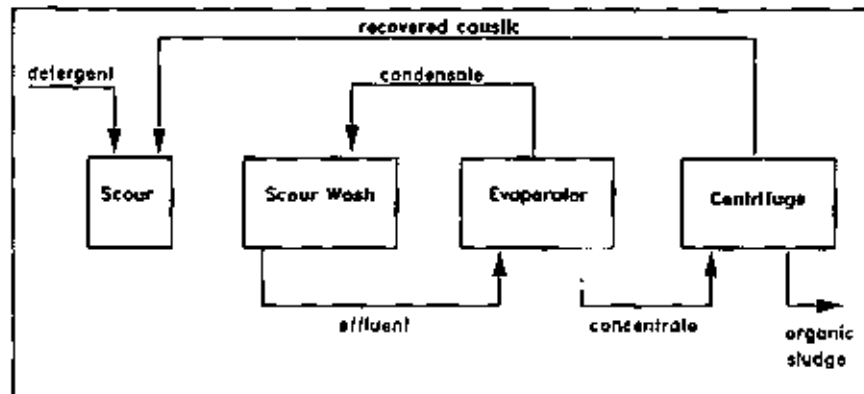
In these two latter processes, the anodic gas generated in the membrane electrolysis cell is used to neutralise incoming effluent.

5.1 Evaporation and Centrifugation of Strong Scour Effluents (Appendix 4)

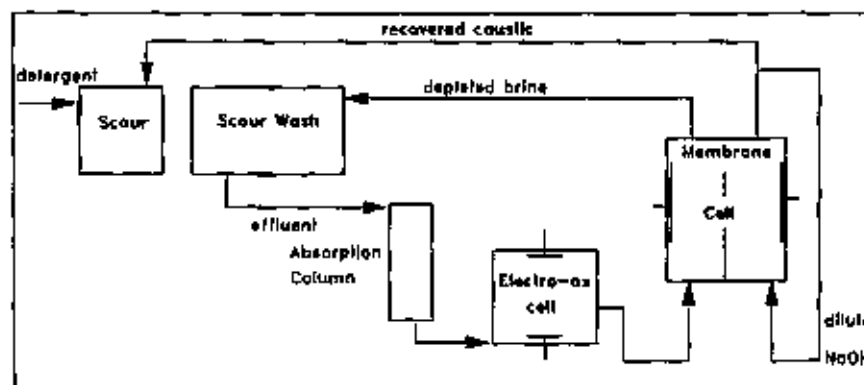
A possible method of recovering caustic from strong scour effluent for reuse in the scouring process is evaporation.

Laboratory scale evaporation and centrifugation trials were conducted on cotton scour effluent. Evaporation of the effluent resulted in the formation of a sludge which could be separated by centrifugation. The sludge composition was found to be dependent on the degree of evaporation and the sodium hydroxide content of the concentrate.

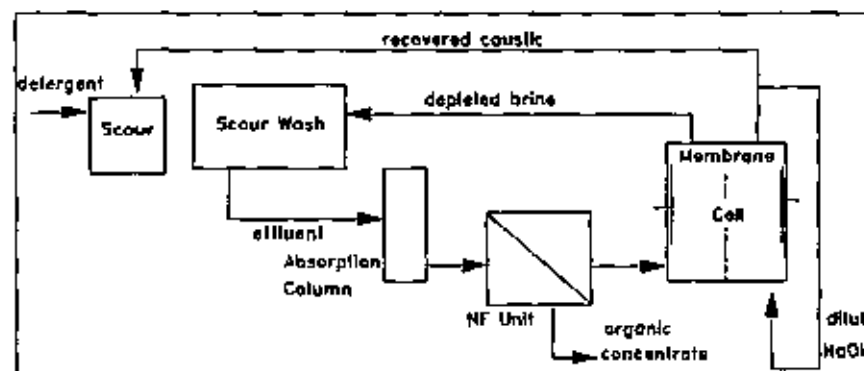
The scour effluent was concentrated to various degrees ranging from 2,5 % to 40 % NaOH. Results indicate that at a concentration of between 25 % and 30 % NaOH, optimal removal of calcium and magnesium species, by their incorporation in the sludge, was obtained. At this concentration 25 % of the soluble organic carbon was also removed.



1a) Treatment by Evaporation



1b) Treatment by Electro-oxidation and Electrolysis



1c) Treatment by Nanofiltration and Electrolysis

FIGURE 1

TREATMENT CONFIGURATIONS FOR STRONG SCOUR EFFLUENT

In most scouring applications concentrations of less than 10 % sodium hydroxide are used. Two options therefore exist :-

- (i) scour effluent may be evaporated to minimum reuse concentration,
- (ii) scour effluent may be concentrated to 2 to 3 times the required concentration and then diluted before reuse.

The advantage of the latter option is that impurity removal is maximised, resulting in a better quality reclaimed caustic. However, economic considerations associated with operation of an evaporator at high water recoveries, would have to be evaluated.

Laboratory tests were undertaken to evaluate the effect that the presence of sizing chemicals had on the quality of caustic reclaimed by evaporation. Two effects were noted :-

- (i) a viscous gel was formed on cooling of the evaporator concentrate (even at low water recoveries). This gel was separated very slowly from solution by filtration.
- (ii) The removal of calcium ions, magnesium ions, and soluble organic carbon from the evaporator concentrate was twice as great as achieved by the evaporation of size-free scour effluent.

A series of five pilot-plant trials using a falling-film evaporator were conducted.

Scour effluent containing approximately 1.6 % sodium hydroxide was concentrated to approximately 10 % sodium hydroxide.

The results of the trials indicated that :-

- (i) fouling of the heat exchange surfaces was negligible,
- (ii) foaming occurred but was reduced to satisfactory levels by the addition of anti-foam and the replacement of a centrifugal pump with a Mono pump.
- (iii) the condensate produced was of sufficient quality to be reused as process water in the scour rinse range.
- (iv) prefiltration to remove lint and other suspended matter was necessary,
- (v) the scour concentrate was of sufficient strength and quality for reuse in the scouring process.

Three trials were conducted using reclaimed caustic in the scouring process. Although the reclaimed caustic contained two to three times more organic matter than virgin caustic, no interference with the scouring action of the sodium hydroxide on the cotton impurities occurred and the final cloth quality was not impaired.

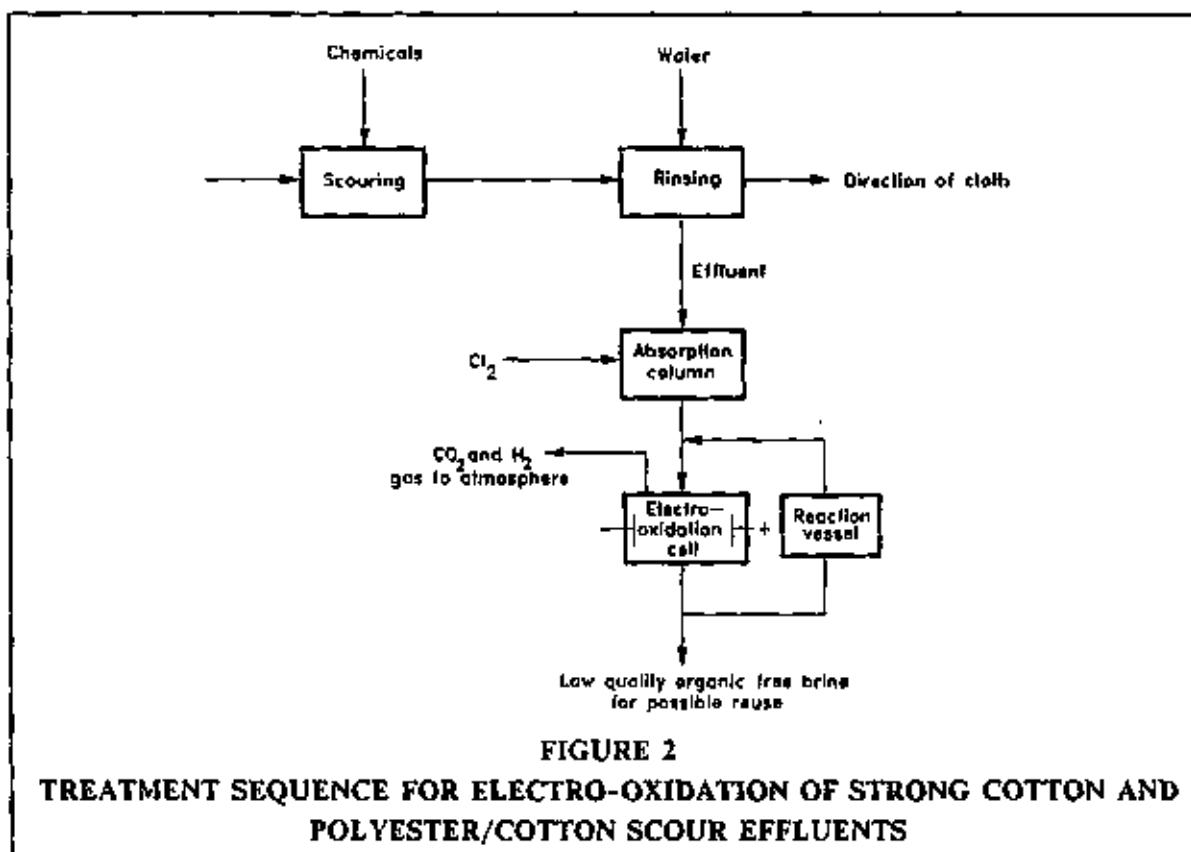
The cloth scoured in reclaimed caustic was subsequently printed and dyed as part of a normal production run. No problems were encountered with further processing and the cloth passed all quality control tests.

5.2 Electro-oxidation of Strong Cotton and Polyester/Cotton Scour Effluents (Appendix 5)

In order to reduce the organic pollution load of strong cotton and polyester/cotton scour effluent a treatment sequence consisting of :-

- (i) neutralisation of the sodium hydroxide in the effluent using chlorine to produce sodium chloride;
- (ii) electrolysis of the sodium chloride solution in an electro-oxidation cell, without a separator, to generate the oxidation agent, hypochlorous acid

was devised and is shown in Figure 2.



Results of laboratory experiments indicated that graphite and stainless steel anodes are unsuitable and that Dimensionally Stable Anodes (DSA) are the most effective anode materials investigated.

Major causes in reduced efficiency in the removal of COD were the side reactions which can occur in the electro-oxidation cell, producing oxychlorides from hypochlorites. These oxychlorides were not capable of lowering the COD of the effluent. Conditions which favoured the formation of oxychlorides were :- high temperature, low chloride concentration, high hypochlorite concentration and a high current density. Operation at a current density of 1900 A/m^2 and a low stirring rate enabled 60 % of the COD to be removed at 100 % current efficiency. The oxidation products of the COD removal were carbon dioxide and water ; the carbon dioxide/carbonate/bicarbonate system buffered the effluent at a pH value of about 9.

An electro-oxidation cell (electrode area 0.1 m^2) was constructed and investigations showed that maximum efficiency in the removal of COD from the effluent was achieved when the average pH of the effluent was 5.4. Under these conditions 85 % of the COD was oxidised at 85 % current efficiency. Complete colour removal from the effluent was achieved at all pH values.

5.3 Nanofiltration of Strong Scour Effluents (Appendix 6)

Nanofiltration or charged ultrafiltration is a pressure driven membrane separation process which lies between ultrafiltration and reverse osmosis. Nanofiltration membranes reject organic molecules and bivalent ions while allowing the passage of monovalent ions. These membranes were of particular interest for the treatment of chlorinated scour effluent as preliminary work showed that calcium and magnesium ions could be separated from the effluent with little loss of sodium ions. This would be advantageous as alkali earth ions could cause irreversible damage to the ion exchange membranes in a chlor-alkali cell.

Laboratory experiments were conducted in an attempt to maximise calcium and magnesium rejection during nanofiltration of pretreated scour effluent. The effect of the addition of sequestering agents to chelate "free" hardness ions was investigated and is summarised in Figure 3. EDTA gave the best results, the addition of 30 mg/L reduced the total hardness ions in the permeate by 50 % to 3 mg/L.

During two experimental runs on prechlorinated scour effluent, fouling of the spiral wrap FilmTec FT40 nanofiltration membrane (area = 0,56 m²) was observed with consequent flux decline. Chemical balances over the nanofiltration unit suggested that precipitation of magnesium hydroxide and/or magnesium carbonate occurred, resulting in physical blockage of the membrane.

Permeate recoveries of 90 % were achieved. Over 87 % of the sodium chloride present in the feed was contained in the composite permeate. Hardness ion rejections were between 50 and 90 %.

Where the nanofiltration feed is a sodium carbonate solution as opposed to a sodium chloride solution then careful consideration of the carbonate/bicarbonate/carbon dioxide equilibrium is required in order to maximise sodium salt recovery during nanofiltration. Laboratory scale investigations were conducted to determine the rejection characteristics of the nanofiltration membrane on the aqueous sodium carbonate system.

Experimental results can be explained by the theoretical consideration of equilibrium data (Figure 4). At high pH values, carbonate ions predominate. Since the membrane preferentially rejects bivalent ions, permeability of the ionic species was minimal. In addition, sodium ion rejection was high in order that charge balance across the membrane be maintained.

Minimum rejection of ionic species was observed in the pH range 8,0 to 8,5. At these pH values the predominant species is the monovalent bicarbonate ion, to which the membrane is permeable. Thus maximum recovery of the sodium salt may be achieved by nanofiltration in the pH range 8,0 to 8,5. Membrane fluxes were found to be dependent on solution pH and were lowest at pH values above 9,0. In addition, the concentration of the salt solution influenced rejection : rejections of sodium ions by the membrane were higher at low salt concentrations.

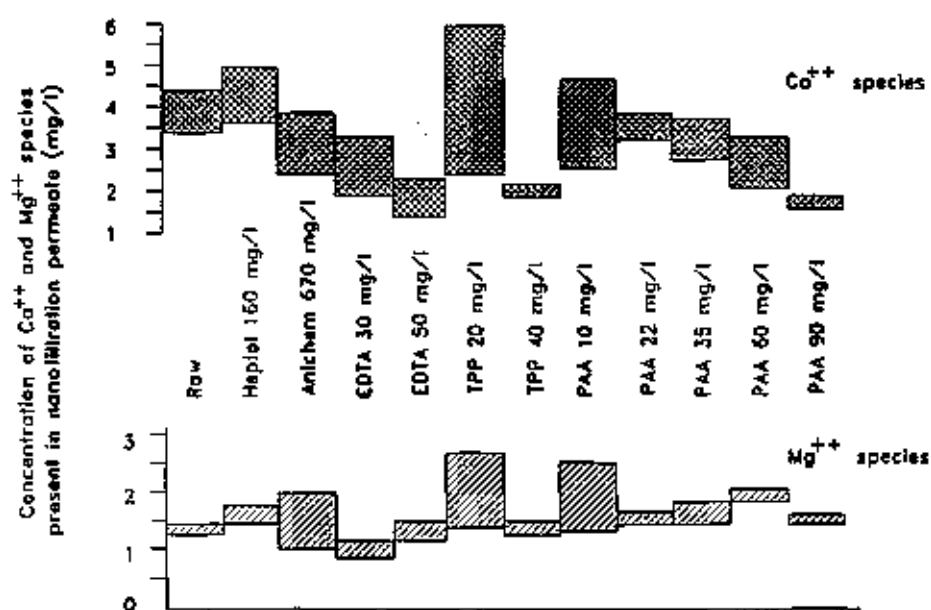


FIGURE 3
EFFECT OF ADDITION OF SEQUESTRANTS ON PERMEABILITY OF
NANOFILTRATION MEMBRANE TO HARDNESS IONS

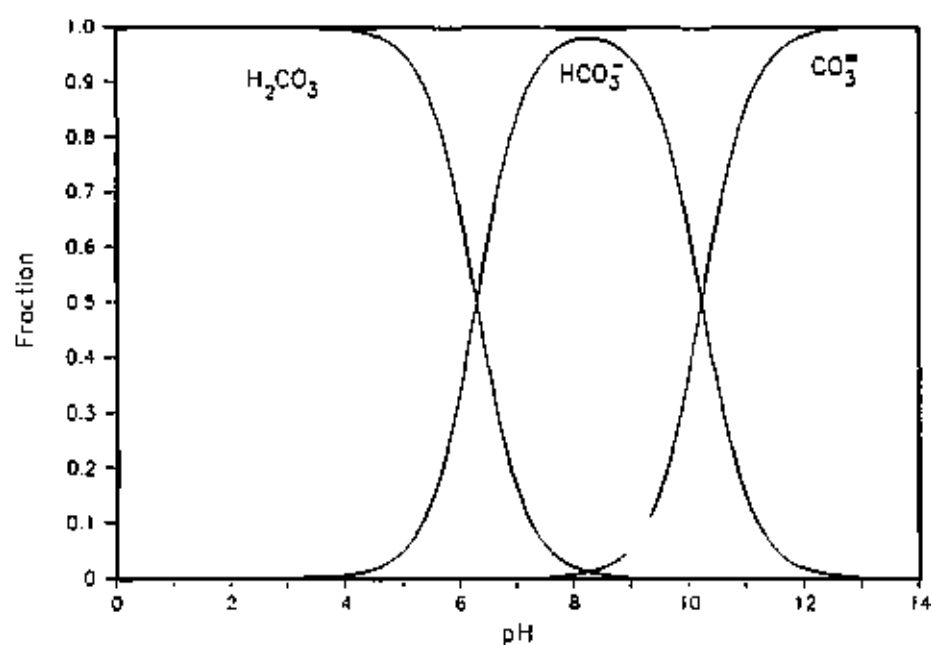


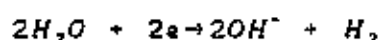
FIGURE 4
DISTRIBUTION OF CARBONATE SPECIES WITH pH

5.4 Electrolytic Recovery of Sodium Hydroxide from Strong Scour Effluent

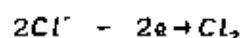
The recovery of sodium hydroxide from scour effluent (pretreated by neutralisation and nanofiltration) in an electrolytic membrane cell was investigated.

Two possible electromembrane processes for the recovery of sodium hydroxide from pretreated scour effluent were developed (the chlorine and the carbon dioxide processes).

In both processes sodium ions are transported, under the influence of an electric current, through a cation exchange membrane into the catholyte. Here sodium hydroxide is formed by the combination of the transported sodium ions with hydroxide ions formed during the electrolytic decomposition of water at the cathode :-

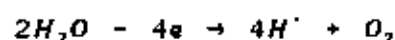


The difference between the two processes lies in the anodic reaction. In the chlorine process the anolyte is sodium chloride and chlorine gas is evolved at the anode :-



The chlorine gas evolved is used to neutralise incoming scour effluent.

In the carbon dioxide process the anolyte is a sodium bicarbonate/sodium carbonate solution. The anodic reaction is the electrolytic decomposition of water with the evolution of oxygen :-



This reaction produces hydrogen ions which lowers the anolyte pH, driving the carbonate-bicarbonate-carbon dioxide equilibrium in the direction which releases carbon dioxide gas from the anolyte. This carbon dioxide gas is used to neutralise incoming scour effluent.

The advantages of the carbon dioxide process are :-

- (i) carbon dioxide is non-corroding and not as great a health hazard as is chlorine. Low cost materials of construction could be used.
- (ii) the capital cost of a cell and electrodes for oxygen generation would be much lower than that for chlorine generation.

The main disadvantage associated with the carbon dioxide process is that carbon dioxide gas is non-oxidising and minimal COD or colour removal from the scour effluent would be achieved during neutralisation.

Laboratory investigations using 3 000 mm² of Nafion 324 cation-exchange membrane were conducted on chlorine pretreated scour effluent. Results showed that half the sodium present in the anolyte could be transferred through the membrane at 100 % current efficiency and all the sodium could be transferred at 50 % current efficiency. As the sodium ion concentration in the anolyte decreased, the current efficiency was reduced.

In a similar laboratory investigation on carbon dioxide pretreated scour effluent a current efficiency of 75 % for the production of sodium hydroxide was averaged.

Semi-technical scale investigations were conducted in one cell of a monopolar stack comprising one anode and one cathode compartment, both constructed from titanium. A mild steel cathode and dimensionally stable anode were used. The division between the anode and cathode chambers was provided by a Nafion 324 cation permeable membrane of area 0,1 m².

The scour effluent used in the investigations contained approximately 25 g/l NaOH. It was pretreated by chlorination and nanofiltration. Analar grade sodium chloride was added to the effluent prior to electrolysis to enable results over a wide range of concentrations to be obtained.

Current efficiencies for the recovery of caustic were 60 to 70 %. The power consumption per ton of 100 % caustic was 3 008 kWh. This figure compares favourably with typical data for recent commercial chlor-alkali membrane cells where the energy consumption is quoted as 2 700 kWh/ton of 100 % NaOH.

During these tests the calcium and magnesium concentrations in the anolyte were approximately 30 times the maximum concentration recommended by the membrane manufacturer. Results indicated no tendency of these ions to migrate from the anolyte during electrolysis and deposit within the membrane causing increased electrical resistance of the membrane. This reinforces the postulation that these ions are chelated to low molecular mass organics to form neutral molecules which do not migrate in an electric field.

5.4.1 Fouling of Electrolytic Cation-exchange membranes by Calcium and Magnesium Salts

The possible fouling of the cation-exchange Nafion 324 membrane by calcium and magnesium ions was investigated in detail using the following systems :-

- (i) pure sodium chloride solution,
- (ii) sodium chloride solutions spiked with calcium and magnesium ions.
- (iii) spiked sodium chloride solutions (ii) containing sequestrants.
- (iv) pretreated strong scouring effluent containing appreciable amounts of calcium and magnesium ions.

Experimental and analytical data obtained during electrolysis of the various solutions were used to calculate the increase in membrane electrical resistance which occurred as a result of fouling by precipitation of calcium and magnesium compounds.

Results showed that :-

- (i) relatively low salt concentrations and/or high calcium and magnesium concentrations in pure sodium chloride anolytes resulted in increased membrane resistance.
- (ii) the addition of sequestrants to spiked sodium chloride anolytes did not control fouling by calcium and magnesium ions.

- (iii) the electromembrane was observed to exhibit very low fouling tendencies toward caustic scour effluent which had been pretreated by chlorination and/or nanofiltration despite relatively high calcium and magnesium ion concentrations. The high stability of the organo-metallic complexes formed during scouring could be responsible for this limited fouling.

6 **AN INTEGRATED SYSTEM FOR THE TREATMENT OF STRONG CAUSTIC SCOUR EFFLUENT : PILOT PLANT INVESTIGATIONS**

The scour effluent from Smith & Nephew, Pinetown, containing potassium hydroxide, and the scour effluent from David Whitehead and Sons, Tongaat, containing sodium hydroxide, were tested using the chlorine system of the treatment sequence. The scour effluent from Da Gama Textiles, King William's Town, containing sodium hydroxide, was tested using the carbon dioxide system of the treatment sequence.

The treatment sequence for pilot-plant experiments was :-

- (i) neutralisation using an acidic gas, either chlorine or carbon dioxide to convert the hydroxide to a neutral sodium (or potassium) salt,
- (ii) cross-flow microfiltration to remove suspended colloidal and waxy contaminants from the neutralised effluent,
- (iii) nanofiltration to separate and recover the neutral sodium (or potassium) salt from the soluble organic and divalent metal contaminants,
- (iv) electrochemical treatment in a membrane cell (Figure 5) to split the sodium or potassium salt solution to form hydroxide, an acidic gas and a depleted salt stream

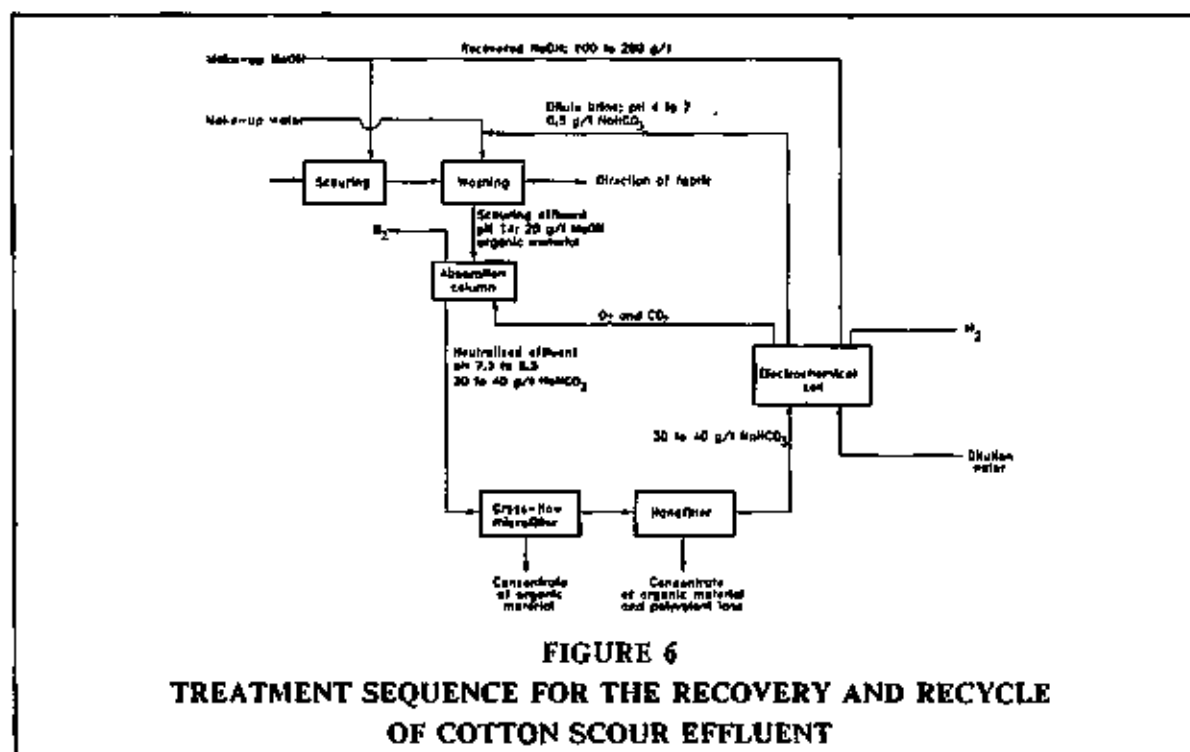
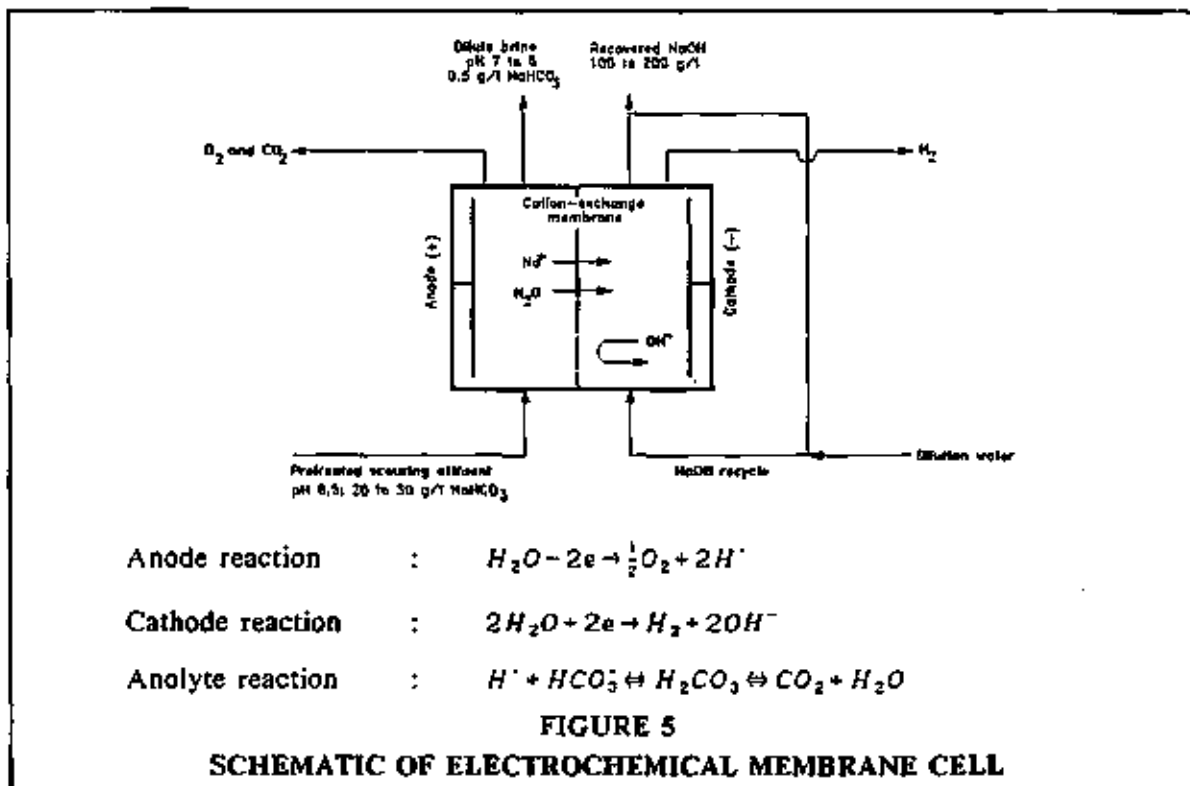
and is illustrated schematically in Figure 6.

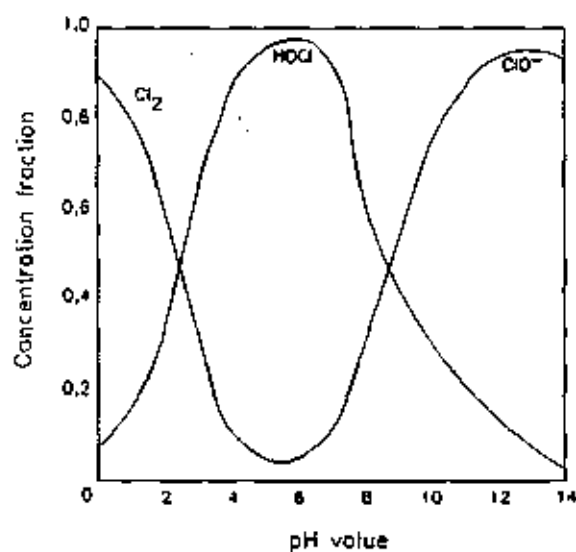
If the acidic gas used in neutralisation is chlorine, then the salt formed is the chloride and the anode process in the electrochemical cell is the oxidation of chlorides to produce chlorine gas which is recycled to neutralisation.

If the anodic gas used in neutralisation is carbon dioxide, then the salt formed is predominantly bicarbonate and the anodic process in the electrochemical cell is the oxidation of water to produce oxygen gas and hydrogen ions. The presence of bicarbonate facilitates a buffering action which results in the evolution of carbon dioxide : $H^+ + HCO_3^- \rightleftharpoons H_2CO_3 \rightleftharpoons CO_2 + H_2O$ which is recycled to neutralisation.

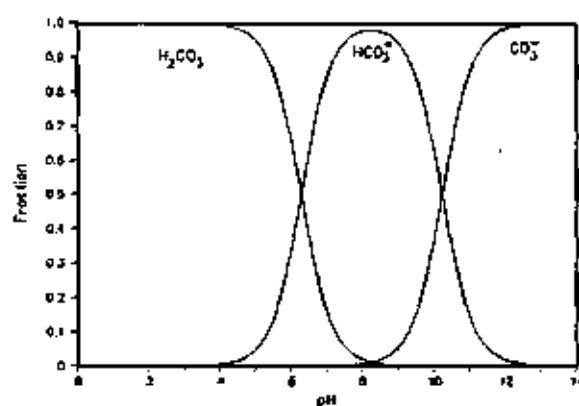
Both processes utilise the pH dependence of the equilibrium which exists between the various forms of chlorine and carbon dioxide in solution (Figure 7a and 7b).

Table 3 compares the chlorine and carbon dioxide systems of the treatment sequence.





a) Chlorine Species



b) Carbon Dioxide Species

FIGURE 7**DISTRIBUTION OF CHLORINE AND CARBONATE SPECIES WITH pH**

The treatment sequence produces the following products :-

- (i) two organic and divalent metal concentrates from the filtration stages, one containing suspended matter and the other soluble contaminants of the scour effluent. These streams comprise 10 % of the initial effluent volume treated.
- (ii) hydrogen and, in the case of the carbon dioxide system, oxygen gases from the electrochemical unit which are vented to the atmosphere. The chlorine and carbon dioxide gases are recycled within the treatment process.
- (iii) a depleted brine solution from the anolyte compartment of the electrochemical unit which could be suitable for recycling to the washing section of the scour process.
- (iv) a pure concentrated caustic stream for recycling to the scour process.

TABLE 3
COMPARISON OF THE CHLORINE AND CARBON DIOXIDE SYSTEMS

Stage	Parameter	Carbon dioxide	Chlorine
Neutralisation	1) acidic gas 2) product salts 3) other effects	CO ₂ NaHCO ₃ Nil	Cl ₂ NaCl, NaOCl oxidation and decolourisation of organics
Nanofiltration	1) chemical addition	Nil	reducing agent
Electrochemical recovery	1) anolyte 2) catholyte 3) anode reaction 4) chemical anolyte reaction 5) cathode reaction 6) gases released 7) materials of cell construction 8) hazards	NaHCO ₃ NaOH $2H_2O - 4e \rightarrow O_2(g) + 4H^+$ $H^+ + HCO_3^- \rightarrow CO_2(g) + H_2O$ $2H_2O - 2e \rightarrow H_2(g) + 2OH^-$ O ₂ , CO ₂ , H ₂ PVC, polypropylene Nil	NaCl NaOH $2Cl^- - 2e \rightarrow Cl_2(g)$ Nil $2H_2O - 2e \rightarrow H_2(g) + 2OH^-$ Cl ₂ , H ₂ PVC, titanium chlorine toxicity explosive

The design of a pilot-plant for both the chlorine and the carbon dioxide systems consisted of an absorption column, a cross-flow microfilter with a tube area of 0,45 to 0,75 m², a nanofilter with a membrane area of 0,37 to 0,56 m² and an electrochemical cell with a total membrane area of 0,1 m². In the chlorine system a single cell constructed from titanium and supplied by Uhde was used. In the carbon dioxide system two cells of a bipolar stack, constructed from PVC and supplied by Steetley Engineering Ltd., was used.

Both pilot-plants were capable of treating 150 t of scour effluent daily with the production of :-

- 3 kg caustic as a 50 to 200 g/l solution.
- 135 t depleted brine.
- 75 g (850 t) hydrogen gas.
- 600 g (420 t) oxygen gas.
- 15 t of organic concentrate.

6.1 Smith and Nephew (Appendix 8, Part 1)

Kiering at this factory is achieved using potassium hydroxide and not sodium hydroxide, since there was a sodium discharge limit in the factory. The elevated temperatures and pressures in the kier vessel mean that caustic concentrations are approximately half of those used in conventional scouring, and hence effluent concentrations are lower.

The effluent streams were characterised to provide an indication of effluent loading. The kier liquor contained 30 g/l of potassium, half of which was present as the carbonate and half as the hydroxide. The kivered cloth is washed batchwise, four times : 85 % of the impurities on the cloth are removed in the first wash and the average water

consumption during washing is high, at 40 l/kg fabric. In order to recover potassium hydroxide a concentration stage (improved washing or reverse osmosis) would be necessary.

All pilot-plant trials were thus conducted using kier liquor and not kier effluent.

Table 4 summarised the composition of the kier liquor in different stages of treatment using the chlorine system. On average, the pretreatment sequence of neutralisation, cross-flow microfiltration and nanofiltration removed all the colour, 60 % of the organics and the chemical oxygen demand, 85 % of the calcium and 25 % of the magnesium.

TABLE 4 EFFECT OF CHLORINE TREATMENT SEQUENCE ON KIER LIQUOR FROM SMITH AND NEPHEW					
Determinand	Composition of process stream				
	Kier liquor	After neutralisation	After microfiltration	After nanofiltration	After electrolysis
pH	14,0	8,5	9,0	9,3	2-6
Conductivity (mS/cm)	91,0	80,0	70,0	60,0	38
Total carbon (g/l)	23,0	20,0	13,0	6,0	6
Inorganic carbon (g/l)	2,0	3,0	3,0	3,0	0
Organic carbon (g/l)	21,0	18,0	8,0	3,0	6
Potassium (g/l)	34,0	31,0	30,0	26,0	13
Sodium (g/l)	2,0	3,0	2,0	3,0	1
Calcium (mg/l)	63,0	85,0	8,0	6,0	10
Magnesium (mg/l)	60,0	70,0	68,0	50,0	50
Hydroxide (g/l)	6,0	-	-	-	-
Carbonate (g/l)	4,0	-	-	-	-
Chloride (g/l)	0,7	21,0	21,0	19,0	17
Chemical oxygen demand (g/l)	42,0	-	-	17,0	17
Total solids (g/l)	-	85,0	76,0	-	-
Note : Recovered KOH : 6 to 10 %.					

The flux during cross-flow microfiltration was low due to the waxy nature of the suspended solids in the kier liquor. Nanofiltration fluxes were high and no fouling was apparent. Potassium hydroxide was recovered in the electrochemical unit at 90 % current efficiency and at an electrical power consumption of 3 000 to 4 500 kWh/ton 100 % KOH (R150 to R200 per ton at an electrical energy cost of R0,05/kWh).

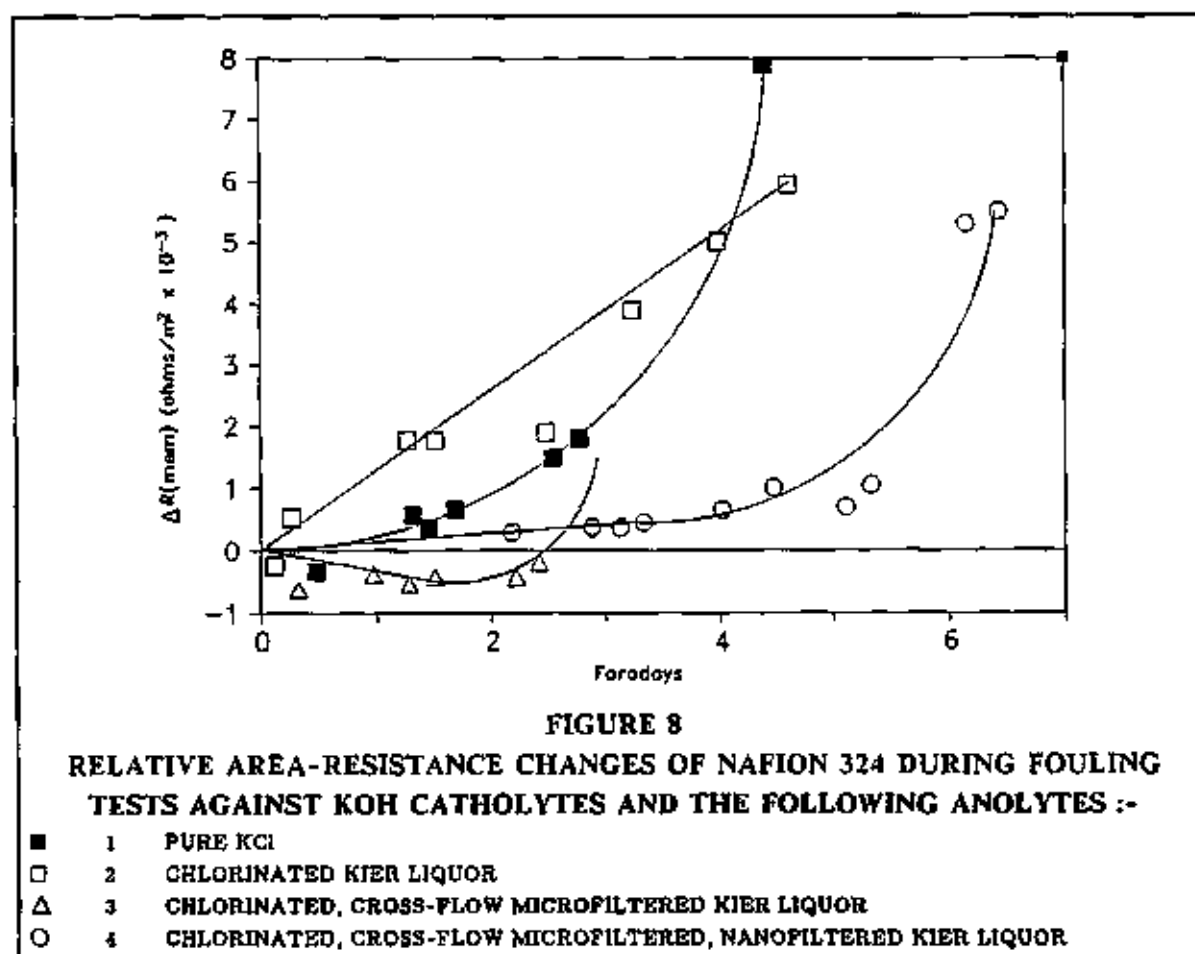
The three major areas of concern were :-

- (i) tests were conducted on kier liquor, reducing its concentration from 30 g/l potassium to 10 g/l potassium. Further recovery of potassium hydroxide was impractical since operational current densities were too low and the electromembrane area requirements of such a system will be excessive.

- (ii) low anolyte concentrations promoted oxygen evolution and dissolution, with subsequent passivation, of the low overpotential precious metal oxide coating on the anode. This resulted in increased operational costs due to :-
- increased anode volt drop and hence increased specific power requirements.
 - decreased chlorine production current efficiency (less than 30 %)
 - anode recoating requirements (1986 quotation : \$2 500 m²).
 - requirements of an independent supply of make-up chlorine to neutralise incoming effluent.
- (iii) the depleted brine solution was very acidic and contained high levels of chloride ions. It was considered unsuitable for reuse in the kier wash range without pH adjustment and without modifications to the existing equipment to ensure that it was corrosion resistant.

The fouling of the electromembrane was investigated in a laboratory two-cell flow-through apparatus.

Figure 8 shows the relative resistance changes of the membrane during fouling tests using four electrolyte combinations.



The nanofiltered sample showed the least fouling tendency, even less than that of analar grade chemicals, despite its elevated calcium and magnesium concentrations. Fouling of the electromembrane by hydroxides of these metals was probably inhibited by organo-metallic complexing in the kier liquor which prevented migration under the application of an electric potential.

6.2 David Whitehead and Sons (Appendix 8, Part 2)

Pilot-plant trials were conducted on the sodium hydroxide scour effluent from David Whitehead and Sons using the chlorine system.

Table 5 summarises the chemical composition of the scour effluent at the different stages of the treatment sequence.

TABLE 5					
EFFECT OF CHLORINE TREATMENT SEQUENCE ON SCOUR EFFLUENT FROM DAVID WHITEHEAD AND SONS					
Determinand	Composition of process stream				
	Scour effluent	After neutralisation	After microfiltration	After nanofiltration	After electrolysis
pH	14,0	8,1	8,5	8,4	1,0
Conductivity (mS/cm)	98,0	55,0	56,0	49,0	50,0
Total carbon (g/l)	11,0	8,0	7,0	7,0	4,0
Inorganic carbon (g/l)	1,0	1,0	1,0	3,0	0,0
Organic carbon (g/l)	10,0	7,0	6,0	4,0	4,0
Sodium (g/l)	17,0	17,0	16,0	17,0	9,0
Calcium (mg/l)	16,0	16,0	15,0	13,0	13,0
Magnesium (mg/l)	78,0	70,0	20,0	12,0	16,0
Hydroxide (g/l)	17,0	-	-	-	-
Carbonate (g/l)	2,0	-	-	-	-
Chloride (g/l)	0,6	18,0	17,0	20,0	14,0
Chemical oxygen demand (g/l)	60,0	27,0	22,0	12,0	12,0
Total solids (g/l)	51,0	55,0	52,0	-	-
Suspended solids (g/l)	-	2,0	0,0	0,0	0,0

Note : Recovered NaOH: 10 to 20 %.

Chlorination oxidised the effluent, removing all colour and lowering its chemical oxygen demand by an average of 30 %. The rejection of calcium and magnesium by the cross-flow microfilter was found to be dependent on the pH of the effluent : at high pH values, magnesium was preferentially rejected, at low pH values, calcium was preferentially rejected. Approximately 90 % of the sodium salt was recovered during the pretreatment sequence.

Cross-flow microfiltration fluxes were low and nanofiltration fluxes were high with no long term fouling of the membrane.

During electrolysis of the nanofiltrate in the electrochemical membrane cell, sodium hydroxide was produced at greater than 80 % current efficiency and chlorine at 12 to 32 % current efficiency. The specific power consumption during the cell operation was 3 300 to 4 500 kWh/ton 100 % NaOH (R165 to R225 assuming an electrical energy cost of R0.05/kWh).

Operational current density was strongly dependent on anolyte concentration. Low current densities made it impractical to remove more than 55 % of the sodium from the nanofiltrate.

The advantages of either concentration of the scour effluent before treatment or operation of a background concentration closed-loop wash water recycle system were evident and include :-

- (i) higher operational current densities, thus a significant reduction in capital costs,
- (ii) higher chlorine production current efficiencies which would eliminate the need to purchase make-up chlorine,
- (iii) prolonged anode life,
- (iv) retardation of the anodic water oxidation reaction, which would in turn result in the production of a depleted brine solution which would be far more suitable for reuse in the scour wash process since its pH would be approximately 4 (and not pH 1).

6.3 Da Gama Textiles (Appendix 8, Part 3)

Pilot-plant trials were conducted on the sodium hydroxide scour effluent from Da Gama Textiles using the carbon dioxide system.

Table 6 summarises the scour effluent composition after each stage in the treatment sequence.

The pretreatment sequence neutralised the effluent and removed, on average, 86 % of the COD, all the colour, 65 % of the organics and calcium and 50 % of the magnesium from the scour effluent.

The electrochemical stage of the pilot-plant trials recovered sodium hydroxide at an average current efficiency of 62 % and at an average power consumption of 6 897 kWh/ton 100 % NaOH. The sodium hydroxide stream was extremely pure and the depleted brine stream was of a suitable quality for reuse in the scour wash process without pH adjustment.

A problem experienced during the trials was the uneven electrolyte flow to the membrane cells resulting in :-

- (i) gas blinding and increased volt drop which increased the power consumption by 30 to 40 %.
- (ii) non-uniformity of current distribution, resulting in localised polarisation and subsequent loss in current efficiency.

- (iii) poor membrane positioning in the cell, resulting in membrane-anode contact, non-uniform current distribution, lowered current efficiency and mechanical abrasion of the precious metal oxide coating on the anode surface.

TABLE 6
EFFECT OF CARBON DIOXIDE TREATMENT SEQUENCE ON SCOUR EFFLUENT
FROM DA GAMA TEXTILES

Analysis	Raw scour effluent	After neutralisation	After CFMF	After NF	After electrolysis	
					Brine	NaOH
pH	13,5	8,6	8,4	9,0	6,2	14,0
Conductivity (mS/cm)	6,4	2,4	2,5	2,3	0,2	-
Total carbon (g/l)	4,0	7,9	7,8	5,9	0,4	-
Inorganic carbon (g/l)	0,3	4,3	4,6	5,2	0,0	-
Organic carbon (g/l)	3,7	3,6	3,0	0,7	0,4	-
Chemical oxygen demand (g/l)	8,3	8,3	5,3	0,5	0,5	-
Hydroxide (g/l)	4,1	0,0	0,0	0,0	0,0	70,0
Carbonate (g/l)	2,6	1,9	2,0	3,4	0,0	1,5
Bicarbonate (g/l)	0,0	16,1	16,5	11,5	0,0	0,0
Sodium (g/l)	8,4	8,2	8,8	7,2	0,3	97,0
Calcium (mg/l)	45,0	45,0	23,0	15,0	4,0	-
Magnesium (mg/l)	7,0	5,0	6,0	3,0	1,0	-
Total solids (g/l)	22,0	22,0	20,0	0,0	0,5	-

Figure 9 shows the relationship between anolyte flow rate and the volt drop across both cells and the cell stack. Volt drop is also dependent on temperature : it was shown that the volt drop, and hence the power consumption could be decreased by 20 % by operation of the cell stack at a constant temperature of 80 °C. In summary, it was predicted that power consumption could be lowered to between 3 500 and 4 000 kWh/ton 100 % NaOH (R175 to R200 assuming an electrical energy cost of R0,05/kWh) if electrolyte flow distribution and rate were controlled and if electrolyte temperature was maintained between 60 and 80 °C.

The limiting current density for the anolyte increased at a rate of 600 A/m² per 10 g/l increase in anolyte sodium concentration (Figure 10).

In order to operate at a high anolyte concentration a background concentration closed loop recycle wash system (Figure 11) was proposed. Scoured fibre is washed with a sodium bicarbonate solution instead of mains water. Only the pick-up sodium will be recovered in the treatment process. The operation of a recycle loop containing 10 to 30 g/l Na⁺ would decrease the required electromembrane area by between 58 and 88 % (Figure 12).

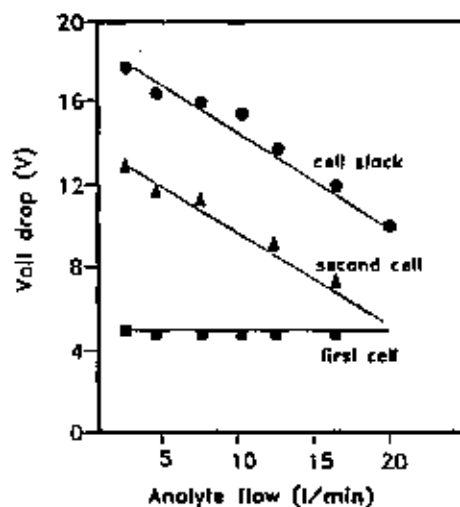


FIGURE 9
RELATIONSHIP BETWEEN ANOLYTE FLOW RATE AND VOLT DROP ACROSS
 ■ FIRST CELL
 ▲ SECOND CELL
 • CELL STACK

- Note :
- 1) series flow configuration
 - 2) catholyte flow rate = 15 l/min
 - 3) electrolyte temperatures = 35 °C
 - 4) operational current density = 1 000 A/m²

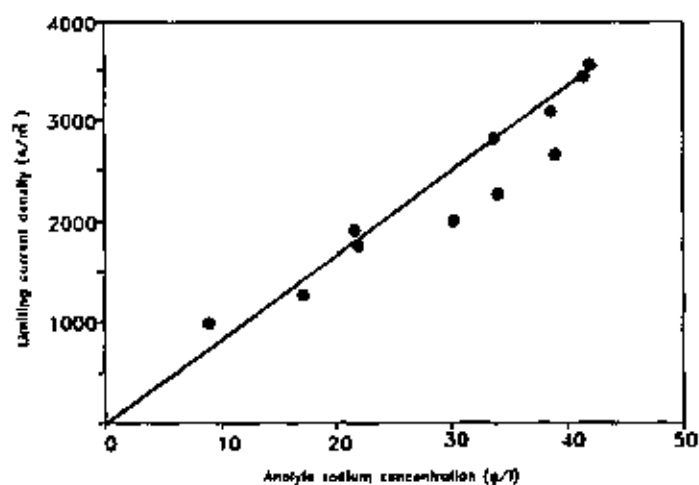


FIGURE 10
RELATIONSHIP BETWEEN LIMITING CURRENT DENSITY AND
ANOLYTE CONCENTRATION FOR ELECTROLYSIS OF PRETREATED
SCOUR EFFLUENT SPIKED WITH SODIUM BICARBONATE

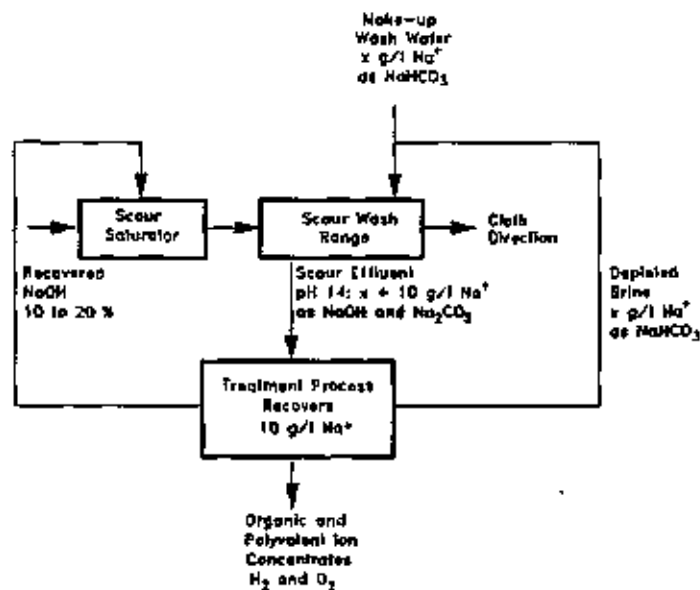


FIGURE 11
SCHEMATIC OF A BACKGROUND CONCENTRATION
CLOSED LOOP RECYCLE WASH SYSTEM

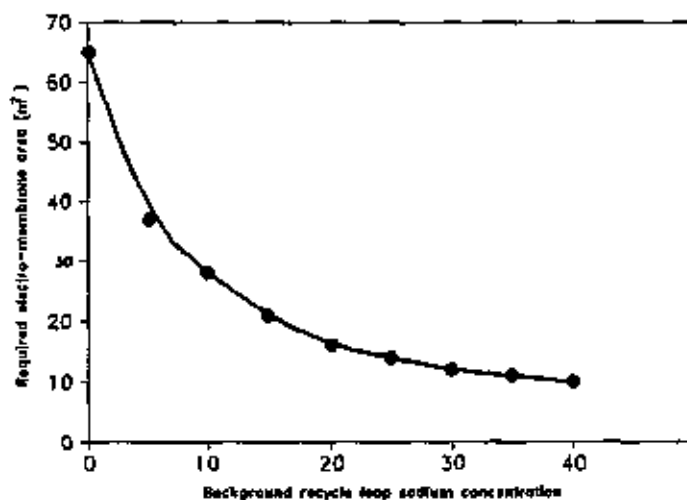


FIGURE 12
RELATIONSHIP BETWEEN ELECTROMEMBRANE AREA AND THE
BACKGROUND SODIUM CONCENTRATION IN THE RECYCLE LOOP
FOR ELECTROLYSIS OF PRETREATED SCOUR EFFLUENT AT :-

- 1) 62 % CURRENT EFFICIENCY
- 2) PRODUCTION RATE = 1 TON PER DAY
- 3) ELECTROLYTE FLOW RATES = 15 L/MIN
- 4) ELECTROLYTE TEMPERATURES = 40 TO 50 °C

The two main factors influencing the economic viability of the process are the anode coating and the membrane life expectancies. Long anode coating life was predicted if mechanical abrasion by the membrane was controlled. No increase in membrane resistance during the pilot-plant trials was observed. A white deposit on the anode surface of the membrane was identified as calcium carbonate which formed in the where localised polarisation had occurred.

6.4 COMPARISON BETWEEN THE CHLORINE AND THE CARBON DIOXIDE SYSTEMS

Pilot-plant trials indicated that the carbon dioxide system was the preferred route since :-

- (i) chlorine gas is a health hazard and under certain conditions, is explosive in the presence of hydrogen gas. Chlorinated organics are formed, the toxicities of which are unknown.

Carbon dioxide is completely non-toxic.

- (ii) indications are that the operating costs of running the chlorine system would be high :-

- chemical addition of reductants is necessary after chlorination to protect the nanofiltration membrane.
- relatively frequently recoating of the precious metal oxide anode surface.
- need for make-up chlorine to compensate for the low anodic current efficiencies for chlorine production.
- chemical addition for neutralisation of the depleted brine before reuse.

In the carbon dioxide process, long anode life is predicted and equal amounts of sodium and carbon dioxide are recovered during electrolysis. The depleted brine solution is suitable for reuse in the scour wash range without pH adjustment, thus providing water savings.

- (iii) the capital costs associated with the chlorine system are high since all equipment must be chlorine resistant and constructed from titanium. If the depleted brine is neutralised for reuse, all processing equipment would need to be replaced with equipment resistant to chloride corrosion.

The carbon dioxide system produces a non-corroding depleted brine and the treatment plant may be constructed from PVC or polyethylene.

- (iv) depletion of the nanofiltrate anolyte during electrolysis using the chlorine system was only partial. Metal ion concentrations could not be decreased below 8 to 12 g/l before operational current densities became impractically low. Hence only 50 to 60 % of the sodium in the scour effluent could be recovered.

In the carbon dioxide system, however, depletion of the anolyte could be achieved down to 0,3 g/l of sodium, at reasonable current densities. This constituted a 90 % recovery of sodium in the effluent.

- (v) If a background concentration wash water recycle loop were to be instituted, it would be more easily accommodated if the background salt were sodium bicarbonate and not sodium chloride, since corrosion of existing equipment would be minimal

7 SUPPLEMENTARY SURVEYS IN THE TEXTILE INDUSTRY

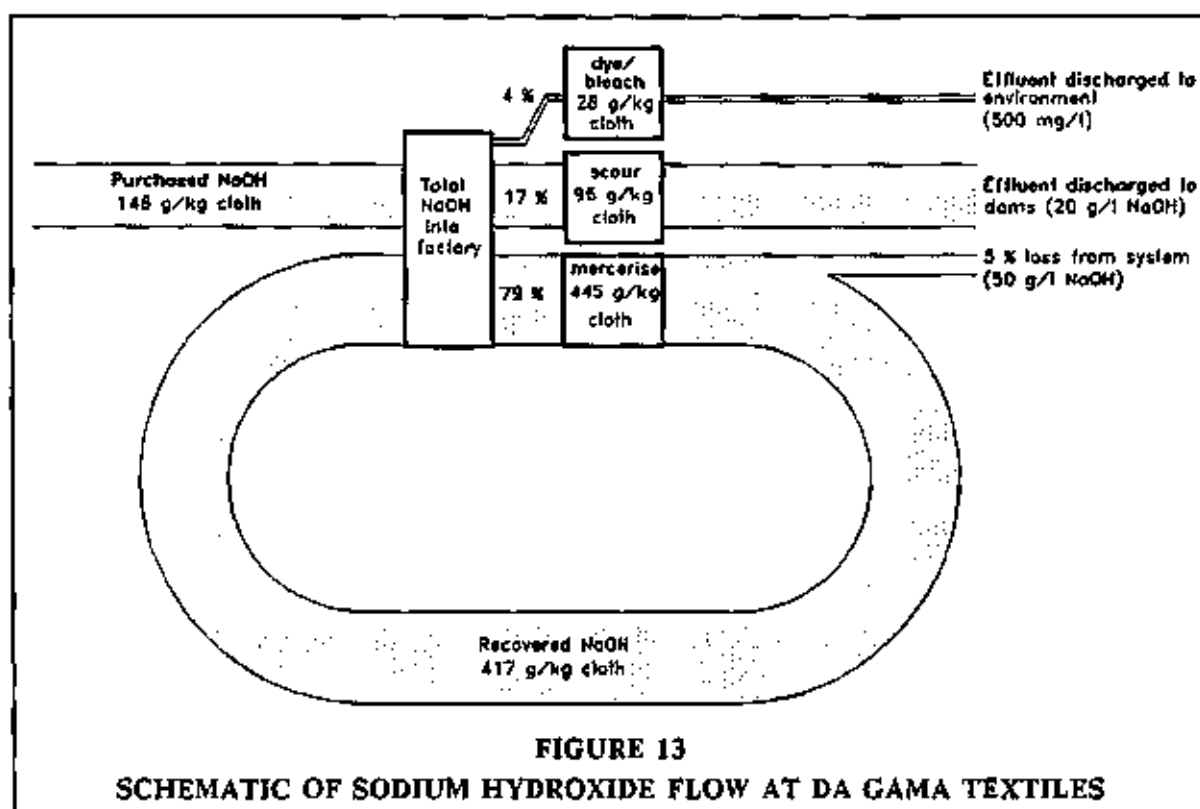
During the course of this project there was an increased awareness by the textile industry for the need for technologies which would ensure both pollution abatement and resource conservation. As a result, the Pollution Research Group was approached by various individual textile factories to carry out detailed pollution, chemical, water and effluent surveys within the factories. The results of the surveys are summarised below :-

- (i) Implementation of recommendations by the Pollution Research Group regarding sodium hydroxide reticulation at David Whitehead and Sons lowered the annual sodium hydroxide cost during 1986 by R1,2 million compared to that of 1985.
- (ii) A detailed pollution and effluent survey was performed at Burhose, Estcourt. From a database which was produced, the quantities of water, dyestuffs and chemicals which should have been consumed were compared to actual usage. Only one third of the metered water could be accounted for. The chemical balance highlighted a large over-use of leveller. Recommendations were made to reduce the over-use of chemicals and dyes which would also reduce the colour in the effluent by 63 % and the OA by 66 %. Subsequently the factory has invested in low pollution load equipment and further savings have been achieved.
- (iii) The effluent load from various textile operations was predicted using previous survey results and advice on pollution prevention techniques was given to a Swaziland textile mill in its planning stages. An in-house sodium hydroxide cascade system was formulated in which mercerising effluent would be used in scouring, bleaching and dyeing and as an ion-exchange regenerant. Emphasis was put on the installation of additional textile processing equipment and not on the installation of effluent treatment equipment e.g. an evaporator was substituted with extra drying cans on the mercerising process. A theoretical water consumption of 30 l/kg fabric was predicted which is one third that of conventional cotton processing mills.
- (iv) A sodium hydroxide survey was conducted at the Consolidated Textiles which indicated where losses were occurring. Recommendations were made regarding techniques to enable minimisation of chemical losses.

In addition, prior to pilot plant trials at Da Gama Textiles, a sodium hydroxide balance across the entire factory was established.

The specific sodium hydroxide usage in the factory is shown in Figure 13. The textile processes consuming sodium hydroxide are :-

- (i) mercerising : 79 % of the total factory usage (445 g/kg fabric). The sodium hydroxide carried over on the fabric after mercerising is washed off and recovered by evaporation.
- (ii) scouring : 17 % of the total factory usage (96 g/kg fabric). The sodium hydroxide carried over on the fabric after mercerising is not recovered, but discharged to solar evaporation dams as a solution containing up to 20 g/l NaOH.
- (iii) dyeing and bleaching : 4 % of the total factory usage (28 g/kg fabric). The sodium hydroxide consumed during these processes is discharged to the environment as streams of low ionic strength.

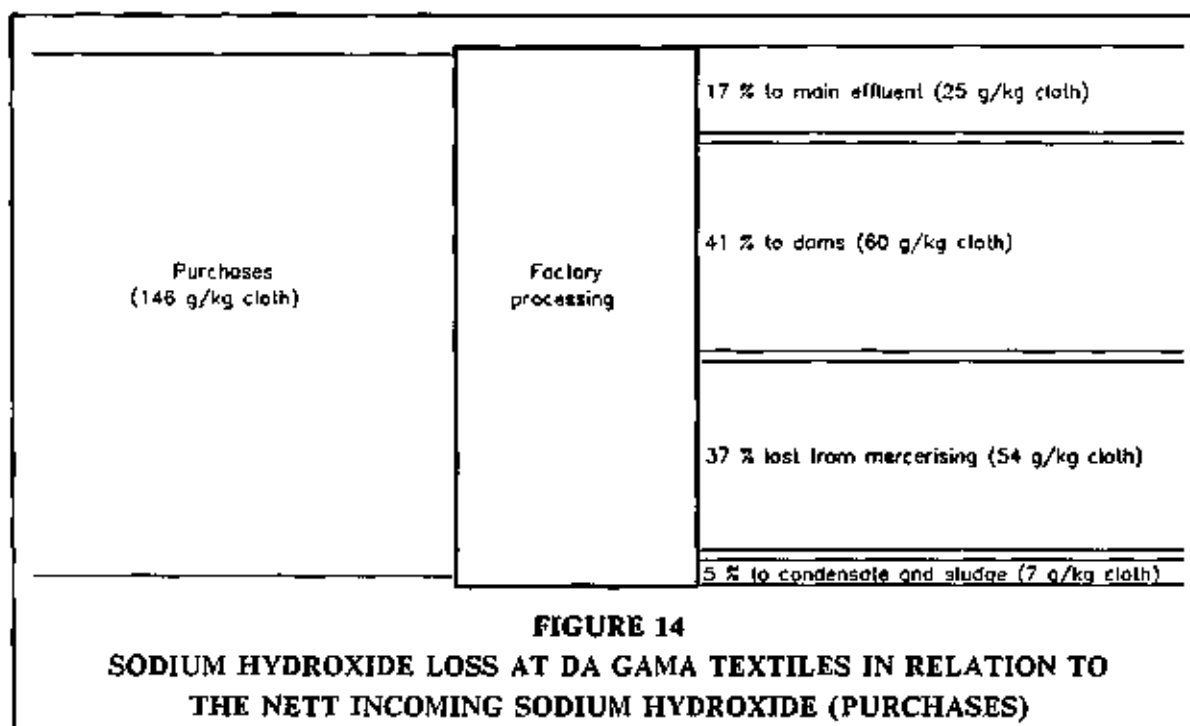


The areas of sodium hydroxide loss were established and are summarised in Figure 14. Of the net sodium hydroxide entering the factory (purchases), 17 % was discharged to the main effluent, 41 % was pumped to the solar evaporation dams and 5 % was lost at the evaporator. The remaining 37 % of the purchases (30 tons/month) were lost from the reticulation system between mercerising and evaporation.

Recommendations were made to enable :-

- (i) elimination of major sodium hydroxide losses,

- (ii) optimisation of the recovery and reuse of sodium hydroxide by the scheduling of all streams,
- (iii) the control of sodium hydroxide usage to reduce processing variables and to achieve maximum consistency in cloth quality.



8 PUBLICATIONS

A total of 6 publications and 9 conference papers have resulted from work undertaken during the course of this project. A South African and USA patent has been granted for the sodium hydroxide recovery process. Patent applications have been lodged in 4 other countries.

8.1 Papers Presented at Conferences

1. Simpson, A.E., Buckley, C.A. and Neytzell-de Wilde, F.G, Caustic Recovery from Bottling Plant, Paper Presented at Coca-Cola Water Management Seminar, Rosebank Hotel, Johannesburg, South Africa, 29th September - 2nd October 1986.
2. Simpson, A.E. and Buckley, C.A., The Recovery and Recycling of Sodium Hydroxide Containing Effluents, Paper Presented at the Institute of Water Pollution Control Biennial Conference, Port Elizabeth, South Africa, 12th - 15th May 1987.
3. Buckley, C.A., Panel Member - Potential for Effluent Treatment and Chemical Recovery, in South Africa Technology Forum on Effluent Treatment and Chemical Recovery by Electrically Driven Membrane Processes, CSIR Conference Centre, Pretoria, South Africa, 29th June 1987.

4. Simpson, A.E. and Buckley, C.A., **The Recovery of Caustic Soda from Caustic Effluents**, Paper Presented at the Technology Forum on Effluent Treatment and Chemical Recovery by Electrically Driven Membrane Processes, CSIR Conference Centre, Pretoria, South Africa, 29th June 1987.
5. Hart, O.O., Simpson, A.E., Buckley, C.A., Groves, G.R. and Neytzel-de Wilde, F.G., **The Treatment of Industrial Effluents with High Salinity and Organic Contents**, Paper Presented at the 3rd World Congress on Desalination and Water Reuse, Cannes, France, 14th - 17th September 1987.
6. Simpson, A.E., Kerr, C.A. and Buckley, C.A., **The Effect of pH on the Nanofiltration of the Carbonate System in Solution**, Paper Presented at the 3rd World Congress on Desalination and Water Reuse, Cannes, France, 14th - 17th September 1987.
7. Simpson, A.E. and Buckley, C.A., **The Treatment of Industrial Effluents Containing Sodium Hydroxide to Enable the Reuse of Chemicals and Water**, Paper Presented at the 3rd World Congress on Desalination and Water Reuse, Cannes, France, 14th - 17th September 1987.
8. Buckley, C.A., Bindoff, A.L., Kerr, C.A., Kerr, A., Simpson, A.E. and Cohen, D.W., **The Use of Speciation and X-ray Techniques for Determining Pretreatment Steps for Desalination**, Paper Presented at the 3rd World Congress on Desalination and Water Reuse, Cannes, France, 14th - 17th September 1987.
9. Simpson, A.E. and Buckley, C.A., **The Recovery and Reuse of Sodium Hydroxide from Industrial Effluents**, Paper Presented at the Symposium on Advances on Reverse Osmosis and Ultrafiltration, American Chemical Society, Division of Industrial and Engineering Chemistry Meeting at the Third Chemical Congress of the North American Continent, Toronto, Canada, 5th - 11th June 1988.

8.2 Publications

1. Simpson, A.E., Kerr, C.A. and Buckley, C.A., **The Effect of pH on the Nanofiltration of the Carbonate System in Solution**, *Desalination*, 64, pp. 305-319, 1987.
2. Buckley, C.A., Bindoff, A.L., Kerr, C.A., Kerr, A., Simpson, A.E. and Cohen, D.W., **The Use of Speciation and X-ray Techniques for Determining Pretreatment Steps for Desalination**, *Desalination*, 66, pp. 327-337, 1987.
3. Hart, O.O., Simpson, A.E., Buckley, C.A., Groves, G.R. and Neytzel-de Wilde, F.G., **The Treatment of Industrial Effluents with High Salinity and Organic Contents**, *Desalination*, 67, pp. 395-407, 1987.
4. Simpson, A.E. and Buckley, C.A., **The Treatment of Industrial Effluents Containing Sodium Hydroxide to Enable the Reuse of Chemicals and Water**, *Desalination*, 67, pp. 409-429, 1987.
5. Simpson, A.E. and Buckley, C.A., **The Recovery of Caustic Soda from Caustic Effluents**, *ChemSA*, pp. 76-80, March 1988.

6. Simpson, A.E., Neytzell-de Wilde, F.G. and Buckley, C.A., The Caustic Recovery from Bottling Plant Effluent, *Water SA*, 14(2), pp. 99-104, April 1988.

8.3 Patents

8.3.1 Granted

1. Buckley, C.A. and Simpson, A.E. (1987) Effluent Treatment, South African Patent No. 87/4406.
2. Buckley, C.A. and Simpson, A.E. (1987) Effluent Treatment, United States of America Patent No. 07/064.339.

8.3.2 Provisional

1. Buckley, C.A. and Simpson, A.E. (1987) Effluent Treatment, Australian Patent No. 74246/87.
2. Buckley, C.A. and Simpson, A.E. (1987) Effluent Treatment, Canadian Patent No. 539.772.3.
3. Buckley, C.A. and Simpson, A.E. (1987) Effluent Treatment, European Patent No. 87205644.4.
4. Buckley, C.A. and Simpson, A.E. (1987) Effluent Treatment, Japanese Patent No. 62.155646.

8.4 Publications Pending

Pollution Research Group, A Guide for the Planning Design and Implementation of Waste Water Treatment Plants in the Textile Industry. PART III: Closed Loop Treatment/Recycle Options for Textile Scouring, Bleaching and Mercerising Effluents, Water Research Commission, Pretoria, 1988. WRC No. TT 48/90. ISBN 0 947447 80 6.

9 LIST OF APPENDICES

APPENDIX 1 : Results and Trials on the Cascading of Process Water in the Preparation of Cotton and Cotton/Polyester Woven Fabrics.

APPENDIX 2 : The Treatment of Carbonising and Bleach Effluents.

Part 1 : Characterisation of Effluent Streams from the Bleach Section at the OTH Beier Plant, Durban.

Part 2 : Laboratory Scale Investigations into the Treatment of Carbonising and Bleach Effluents During the Processing of Raw Wool at the OTH Beier Plant, Durban.

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Part 1 : Laboratory Scale Investigations.

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APPENDIX 4 : Recovery of Caustic from Scour Effluent by Evaporation and Centrifugation.

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APPENDIX 5 : Electro-oxidation of Organics in Cotton Scour Effluent.

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Part 3 : Dependence of Current Efficiency of Electro-oxidation on pH.

APPENDIX 6 : Removal of Organics and Hardness Ions from Prechlorinated Caustic Cotton Scour Effluent by Membrane Separation Techniques.

Part 1 : Preliminary Laboratory Investigations Using Ultra- and Nanofiltration Membranes.

Part 2 : Effect of Sequestering Agents on Hardness Ion Rejection by Nanofiltration : Prechlorinated Scour Effluent.

Part 3 : Semi-technical Scale Nanofiltration Trials : Prechlorinated Scour Effluent.

Part 4 : Rejection Characteristics of a Nanofiltration Membrane in a Sodium Carbonate System.

APPENDIX 7 : The Design of an Electrochemical Pilot-plant for the Recovery of Sodium Hydroxide from Cotton Scour Effluent.

APPENDIX 8 : An Integrated System for the Treatment of Strong Caustic Scour Effluent : Pilot Plant Investigations.

- Part 1 :** The Recovery of Potassium Hydroxide from Kier Wash-off Effluent at Smith and Nephew, Pinetown.
- Part 2 :** Pilot Plant Results for the Electrochemical Recovery of Sodium Hydroxide from Pretreated Scour Effluent from David Whitehead and Sons, Tongaat.
- Part 3 :** The Recovery of Sodium Hydroxide from Scour Effluent at Da Gama Textiles, King William's Town.