Studies on Improved Pulp Productivity by NSSC Pulping of Hardwood

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INTRODUCTION

It has been rightly pointed out, that, because of the limited supplies of coniferous wood, (more than 95% of Indian forest resources being limited to hardwood varieties), and the dwindling supplies of the traditional raw material, Bamboo, the hardwoods would have to form the bulk of future cellulosic raw materials for Indian Pulp and Paper industry (along with agricultural residues.)

And therefore, for meeting the future challenge, it is essential to develop an appropriate Pulping technique having high Pulping productivity (i. e., high yield at equivalent Pulp quality) for the utilization of Indian hardwoods and agri-residues.

Due to its specific delignification action, neutral Sulfite semi-chemical Pulping (NSSC) process has been termed as an appropriate, and efficient process in the manufacture of Pulp and Paper, from hardwoods.

Although some scattered informations are available on the Pulping characteristics of tropical hardwoods by NSSC process, there is a lack of informations pertaining to quantitative relationship amongst the pulping parameters with the delignification reaction rate. With a knowledge of the interrelationship between the different process variables, and the kinetics of the Pulping reactions, the Pulp industry could adopt itself more readily to the wide variety of raw materials, and also achieve a better process control, thus allowing production of more uniform products, possible with higher Pulp productivity at equivalent quality.

Along with the development of an appropriate Pulping process i. e., NSSC Pulping process for the utilization of hardwood at high yield, there is a strong need to develop a techno-economically viable spent Pulping liquor regeneration process, for recycling of Pulping chemicals, along with abatement of pollution hazard from the NSSC Pulping process.

Therefore, a series of R and D Projects were initiated at the Chemical Engineering Department, IIT Bombay, during the last 10 years, with the following objectives :

(1) To evaluate delignification kinetic data along with its correlationship the Pulping variables during NSSC Pulping of tropical hardwoods,

(2) To develop a viable NSSC Pulping liquor regeneration process for improving the economics of the process, along with pollution control,

(3) To evaluate techno-economic feasibility of developing RO/UF based spent liquor treatment system for the disposal of NSSC process effluent for pollution abatement, along with recycling of water/chemicals for reutilization in Pulping, and washing operations.

An attempt has been in the present Work to report our Work relating to the development of a delignification kinetic model for the prediction of Pulping characteristics during NSSC Pulping of trppical hardwoods, and on the technical feasibility of developing a RO/UF based effluent treatment system for the recycling of water/chemicals, along with abatement of pollution hazards.

EXPERIMENTAL WORK

For Pulping studies, Eucalyptus grandis, a fast growing tropical hardwood, was selected as the cellulosic raw material. Initial Pulping experiments were carried out in 250 ml SS-316 autoclave, within the Pulping temperature of 140°C to 180°C, L/M ratio of 4, with 15% chemical charge (consisting of 11% Na_2SO_3

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and 3% Na_2CO_3), heating being carried out in oil bath. Further large scale experiments were carried out in 2 50 litre capacity SS-316 autoclave, under optimal Pulping conditions developed during smaller scale Pulping experiments, and operational data confirmed.

For the estimation of Pulp lignin content at different time intervals, 30 minutes Chlorine Number (CN) method was used 1, and to take heating-up stage during Pulping process in autoclave, equivalent Pulping time was calculated based on the following relationship reported by Chari[2] :

$$t_{eq} = \frac{\int_0^t e^{-E/RT^{-2}}}{e^{-E/RT}}$$

Where T 'is the temperature in at any time t (during heating-up period), and T is the maximum temperature of cook in °K.

Delignification rate expression has been developed based on the assumption that rate of delignification reaction is proportional to the reactant concentration raised to an arbitrary power, i. e.,

$r_L = -dL/dt = K S^a.L^b$, and from Arrhenius law, K may be taken as

$K = K_o e^{-E/RT}$,

where L = lignin concentration, g of lignin in Pulp/100 g of OD wood,

S = Concentration of Na_2SO_3 in cooking liquor, gpl as Na_2O

By applying least square method, a linear regression of % lignin content of Pulp (L) on residual active Na_2SO_3 (S) left in spent Pulping liquor, was obtained as follows :

$$S = I.4622\ L - I8.82IO.$$

For upgrading process economics of NSSC pulping at high yield further experimental studies were carried out on recovery of Pulping chemicals (Na_2SO_3 and Na_2CO_3) from spent sulfite liquor (SSL) in RO/UF set-up. Eeperimental details, and description of the RO/UF equipments are available in our earlier communicatioas[3-4].

Further experiments on RO/UF system were carried out in bigger scale set-up, under optimal operating conditions, evaluated in Test-cell.

After each Pulping run, unbleached pulp was washed properly, Kollerganed, and after proper degree of beating in a Hollander Beater, pulp properties (CN, lignin content, pulp yield) were evaluated.

RESULTS AND DISCUSSIONS

Figures 1, 2, and 3 present the experimental set-up for Pulping Autoclave, RO/UF set-ups for SSL treatm nt systems (smaller and bigger scale experiments).

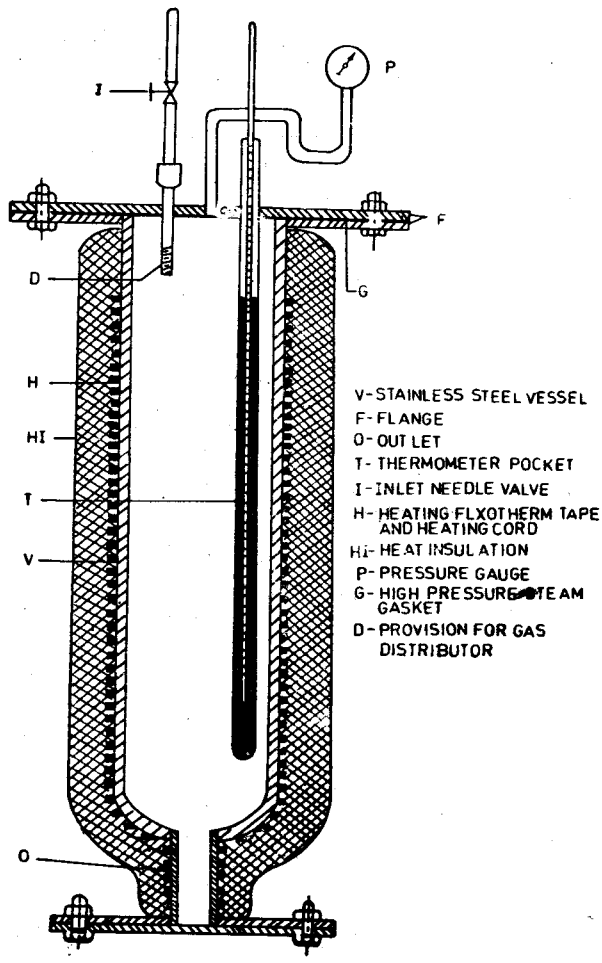


Fig. 1 - STAINLESS STEEL AUTOCLAVE HAVING 2·5 LITRES CAPACITY

During Pulping experiments, total time for Pulping (t) was calculated from $t = t_{eq} + t_c$, where t_c = Pulping time at maximum temperature, and equivalent time was calculated as per the method outlined by Chari[2]. Delignification rate expression has been evaluated based on extensive Pulping studies at differ-

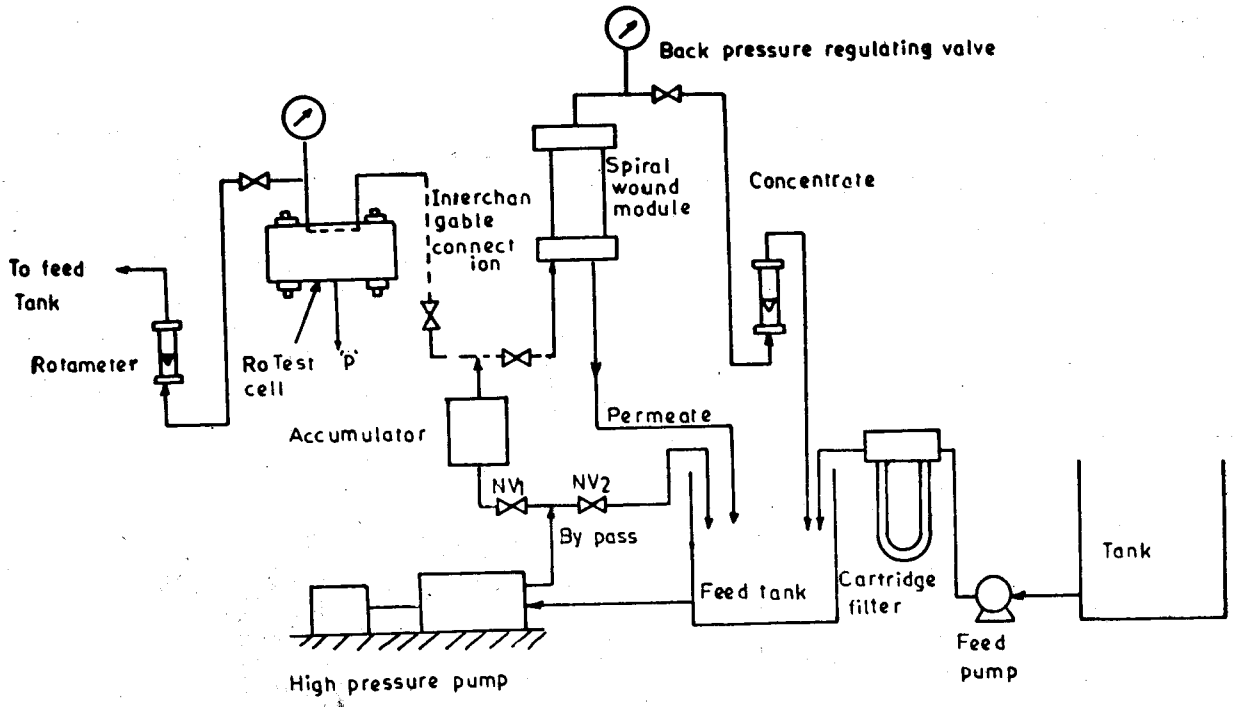
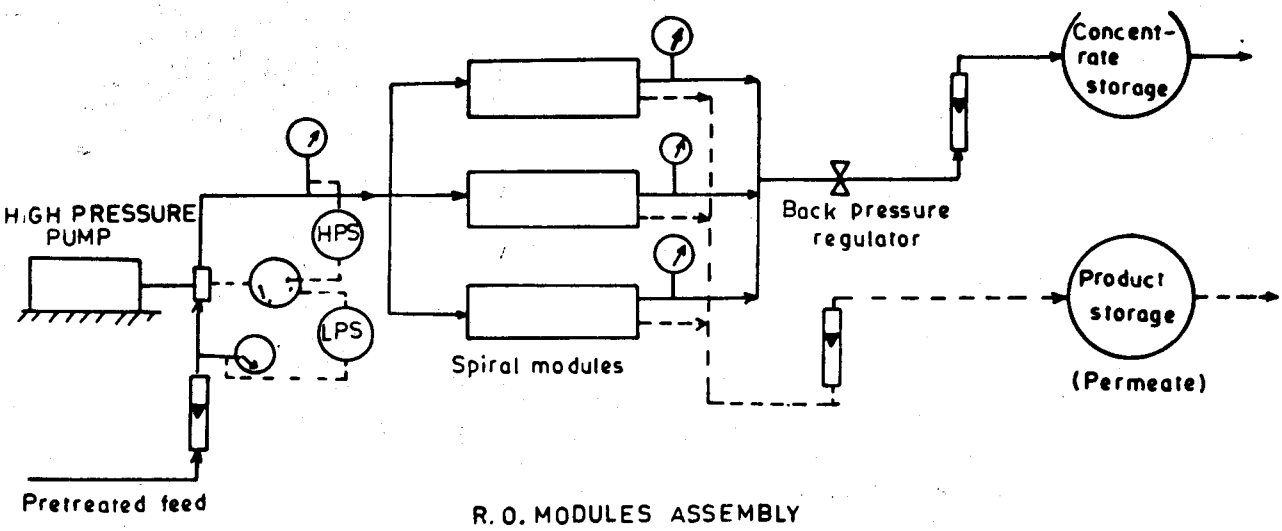


Fig.2-TESTING LOOP FLOW SHEET



R. O. MODULES ASSEMBLY

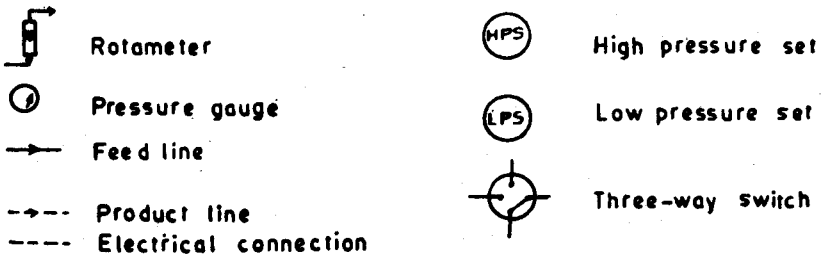


Fig. 3-REVERSE OSMOSIS SYSTEM FLOW SHEET (Large Scale Exprt Set up)

ent temperatures, and the relationship, after simplification assumes the following form :

$$\int_{t_1}^{L_1} \frac{-dL}{(1.4662\ L - 18.821)^a . L^b} = K \int_{t_1}^{t_2} dt$$

where t_1= time in minutes, during which, initial high rate of delignification reaction takes place, L_1= Pulp lignin content at t_1, t_2= Pulp lignin content at any time, t_2.

By trial and error procedure, different values of a and b are assumed, and for each sets of pulping experiments at constant temperature, R. H. S integrated values (B), are plotted as abscissa, L. H.S intergated values as ordinate (referred as A), as per the figures 3, and 4. Results indicate, that, when a= O.25, and b = 1.75, best fit linear relationship of delignification reaction rate with Pulping time, and residual Pulp lignin content at a particular Pulping temperature, is obtained.

Based on K values at different temperature in the form of Arrhenius pl lnk vs I/T, activation energy, and frequency factors have been estimated, and the final rate expression becomes :

$$r_L = dL/dt = 2.07 \times 10^{11} e^{-30,100/RT} . S^{0.25} . L^{1.75}.$$

For evaluating the technical feasibility of RO/UF in the regeneration of spent neutral sulfite spent liquor (SSL), experiments were carried out on permeate flux (Lit/m²/hr) and % solute rejection at different feed pressures, and results are depicted in fig. 5.

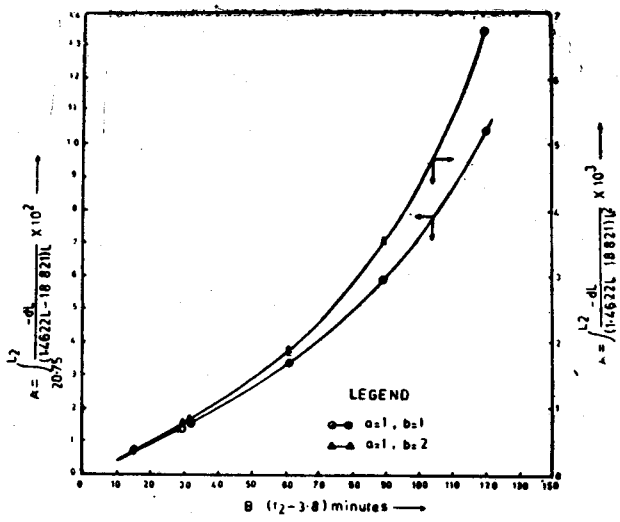
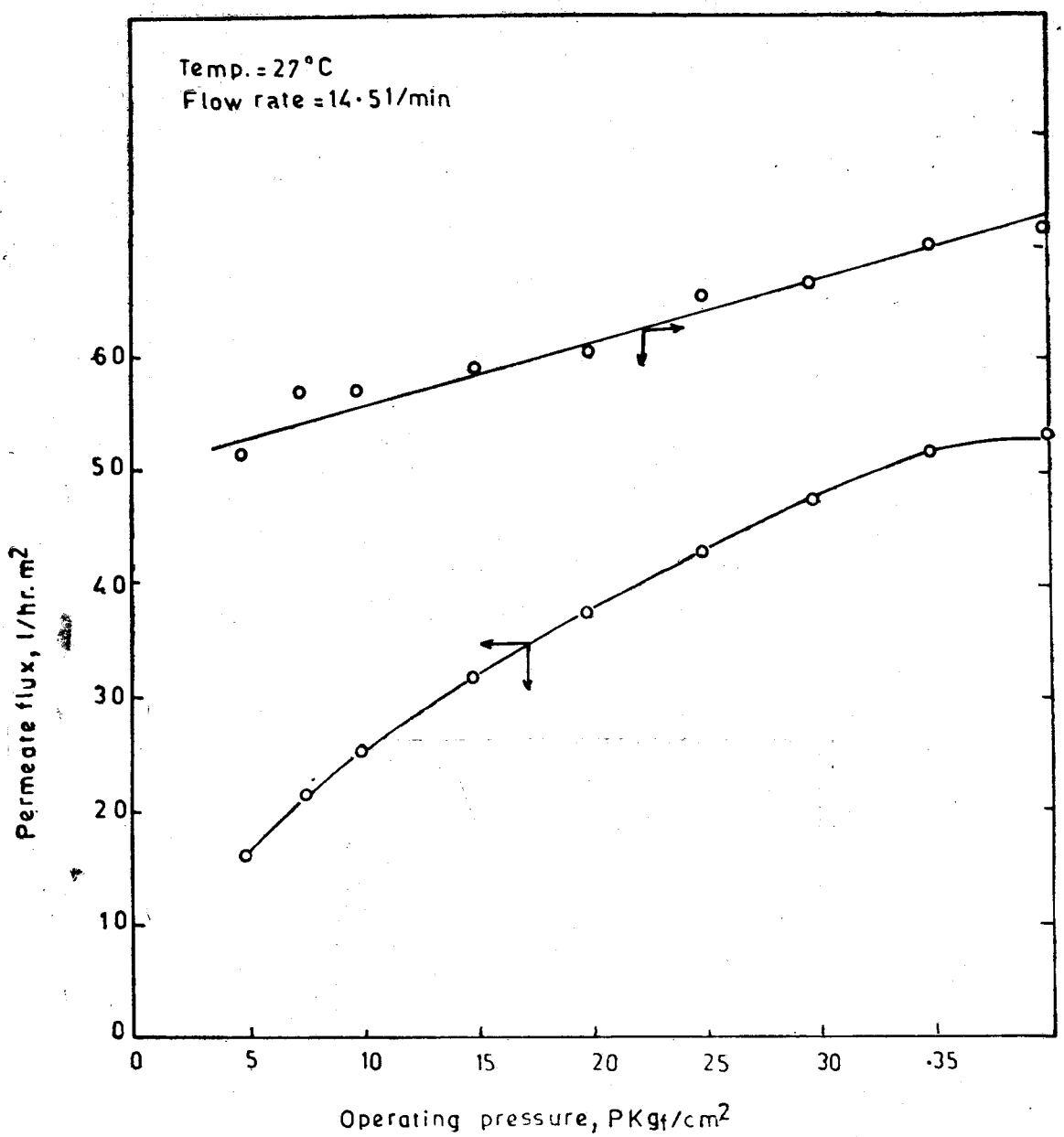


Fig.4 - GRAPHS BETWEEN $\int_{20.75}^{L_2} \frac{-dL}{(1.4662L - 18.821)^a L^b}$ AND $(t_2 - 3.8)$ FOR a=1·0, b=1·0, AND a=1·0, b=2·0 FOR SET OF PULPIN EXPERIMENTS AT 170°C



VARIATION OF PERMEATE FLUX, l/hr. m², AND REJECTION W

OPERATING PRESURE

SYSTEM=Pulp and Paper Mill effluent, 0·675% TDS S Spiral-wound uF membrance (mol wt. cut-off=500) = 2·8 0 m²

For evaluating the quality of the permeate flux (in terms of colour rejection, and active chemical concentracion), further experiments were carried out, and results are presented in fig 6.

With over 92-95% colour rejection, and around 50% of active Pulping chemicals in the form of Na_2SO_3, and Na_2CO_3 in permeate flux at over 80% feed recovery, recycling quality of the permeate flux for Pulping, and Pulp washing is found to be good.

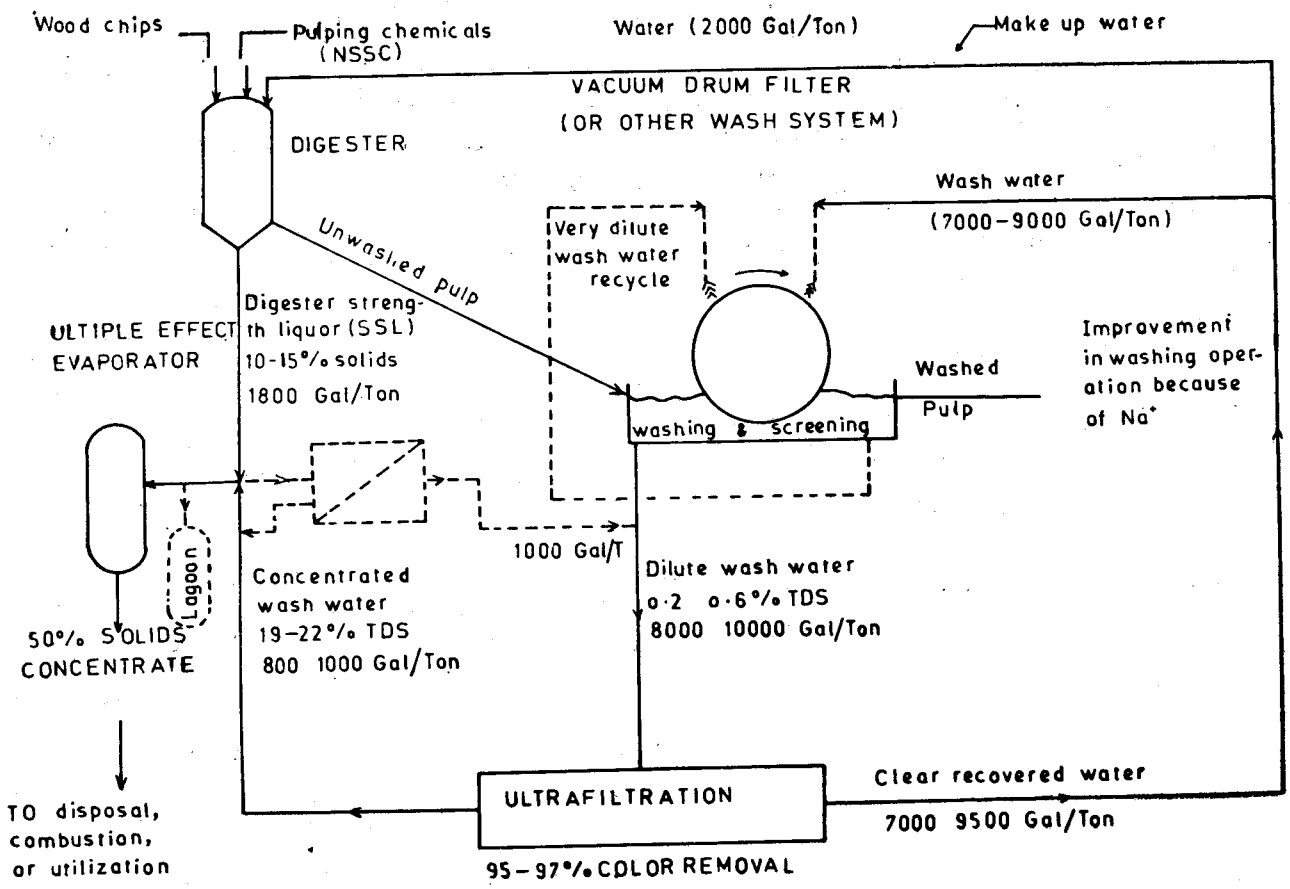


Fig.6-RECYCLING OF SSL WITH POLLUTION CONTROL BY UF

Due to contamination of fouling particulates in SSL feed, permeate flux declined to over 50% of the original flux at time = O, and therefore elaborate feed pretreatment system consisting of clari-flocculation, sedimentation, duel-media sand filtration, and two-stage cartridge filtration (25μ and 5μ), have been designed, and installed. Pretreated SSL feed could give stable permeate flux at about 35 to 40 Lit/M^2/hr at the optimal feed pressure of 15 kg/cm^2, and feed flow rate of 15 lmp, giving colour rejection of over 94% at 80% feed recovery.

The concentrate (about 15 to 20% of the SSL feed) at about 22-25%, obtained according to the proposed RO/UF system, presented in this work, may be disposed off by solar evaporation in lagoon, giving over 75% organic matter (mostly high Mol. Wt. Lignosulfonates and carbohydrate degradation products) in the SSL solids on dry basis.

Extensive Work carried out in USA, and in Scandinavian countries (mostly in Denmark by the DDS Corporation) on the regeneration of SSL by RO/UF, indicates high potentiality of the system, in terms of recovery of purified Lignosulfonates (for multitudes of uses as drilling mud aids, dispersants, adhesives for plywood industry), and sugar products, recyclable water for Pulping and washing operations, along with elimination of pollution hazards considerably, at a energy cost which could be half as compared to the conventional systems of evaporation - incineration routes 5-7.

Further Work is in progress at the IIT-Bombay, on large scale RO/UF studies (according to the system presented in fig. 3), for the evaluation of optimal feed treatment operation (which is very crucial to the sucessful operation of RO/UF Unit on commercial scale), and purification of Lignosulfonates, and active

pulping chemicals, for reutilization in Pulping and other industrial operations.

Since the proposed SSL regeneration system is well suited for straw or bagasse based raw materials (by NSSC Pulping process), it is recommended to carry out Pilot plant scale studies on the appropriateness of the system in Mini Pulp and Paper Industries, which could be designed on the basis of agri-residues as the cellulosic raw materials, and NSSC as the Pulping process.

Over 50% of the total delignification reaction in high-yield NSSC Pulping process (65–75% yield level) is found to take place during the initial heating-up period, and therefore, it is recommended to develop rapid heating NSSC Pulping process, which could give significant amout of increased Pulp yield at equivalent quality.

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