Journal of Scientific & Industrial Research
Vol. 65, February 2006, pp. 169-173

Settling and filtration characteristics of carbonated black liquor from agro based paper mill

S A Mandavgane[1,]* and D Subramanian[2]

[1]Priyadarshini Institute of Engineering and Technology, Nagpur 440 019
[2]Laxminarayan Institute of Technology, Nagpur 440 010

Received 25 May 2005; accepted 18 November 2005

The chemical pulping of agricultural residues poses no problem however major difficulty arises in recovery of heat and chemicals from cooking chemical (black liquor) due to presence of silica in it, at every stage of chemical recovery. Desilication of black liquor is achieved by carbonation effecting delignification. Filtration is a key unit operation in successful desilication technology. Low temperature and low flocculent dose is found to favor sedimentation rate. Filtration of carbonated black liquor using leaf filter shows that temperature favors rate of filtration. Co-precipitation of lignin makes filtration difficult.

Keywords: Black liquor, Desilication, Filtration, Flocculent, Lignin, Sedimentation

IPC Code: G21F9/10

Introduction

Presence of silica in black liquor (BL) obtained from agricultural residues[1] based pulp and paper mill causes serious problems at filtration, washing, evaporation, burning, recausticizing and clarification levels of chemical recovery. Two important criteria for successful desilication[2-8] are selective precipitation of silica and efficient filtration of silica sludge (SS). After selective precipitation, it becomes important to separate SS without much dilution, which will ultimately determine the energy requirement in the evaporator. Thus the extent of dilution becomes one of the important criteria of efficient washing system.

Filtration of SS has been one of the constraints in the development of desilication technology. In SS, lignin also precipitates in the close proximity of desilication and affects the filtration rate. The co-precipitation of lignin invariably and the sludge obtained are reported of coffee brown color[9]. The particle size of silica gels is largely influenced by the temperature and salt concentration[10]. BL, which contains considerable proportion of inorganic and organic salts, will have great influence both on particle size and chemical nature of SS. Lignin salts, which are strongly hydrophilic in nature[11] when co-precipitated with silica, retard the filtration rate.

*Author for correspondence
E-mail: mandavgane@rediffmail.com

The pilot plant at Rakta pulp mills, Alexandria, Egypt employed centrifugal separation of sludge instead of conventional filtering equipment[12] and observed that because of gelatinous nature of SS, the performance was low. Central Pulp & Paper Research Institute (CPPRI), Saharanpur, India and UNIDO[13] who jointly developed demonstration plant capable of processing 40m^3/h of BL at Hindustan Newsprint Ltd. (HNL), Kerala, India, reported that temperature higher than 70^{o}C gives better filtration results whereas precipitated silica particles settles slowly and temperature did not have marked influence on sedimentation. A simple method[14] for precipitating easily filterable acid lignin from kraft BL states that acidification of kraft BL by 2*N* H_2SO_4 to a pH of about 2 in the presence of small amount of organic liquid ($CHBr_3$, CH_2Br_2, CH_2Cl_2, CH_2BrCl, etc) causes precipitation of granular form of lignin, which is quick settling and can be rapidly isolated by filtration. Basu[15] reported that a polyelectrolyte be used as a flocculating agent at the maximum sludge consistency (10%) to achieve an increase in sludge sedimentation rate, consistency and clarification rate.

Precipitated silica has a wide range of industrial applications such as, reinforcement in rubber, leather tanning, to improve ink retention on paper, a pigment and filler in paint and coating, as an abrasive, as an adsorbent and as an electric insulator. Silica as a by-product would contribute to the economics of

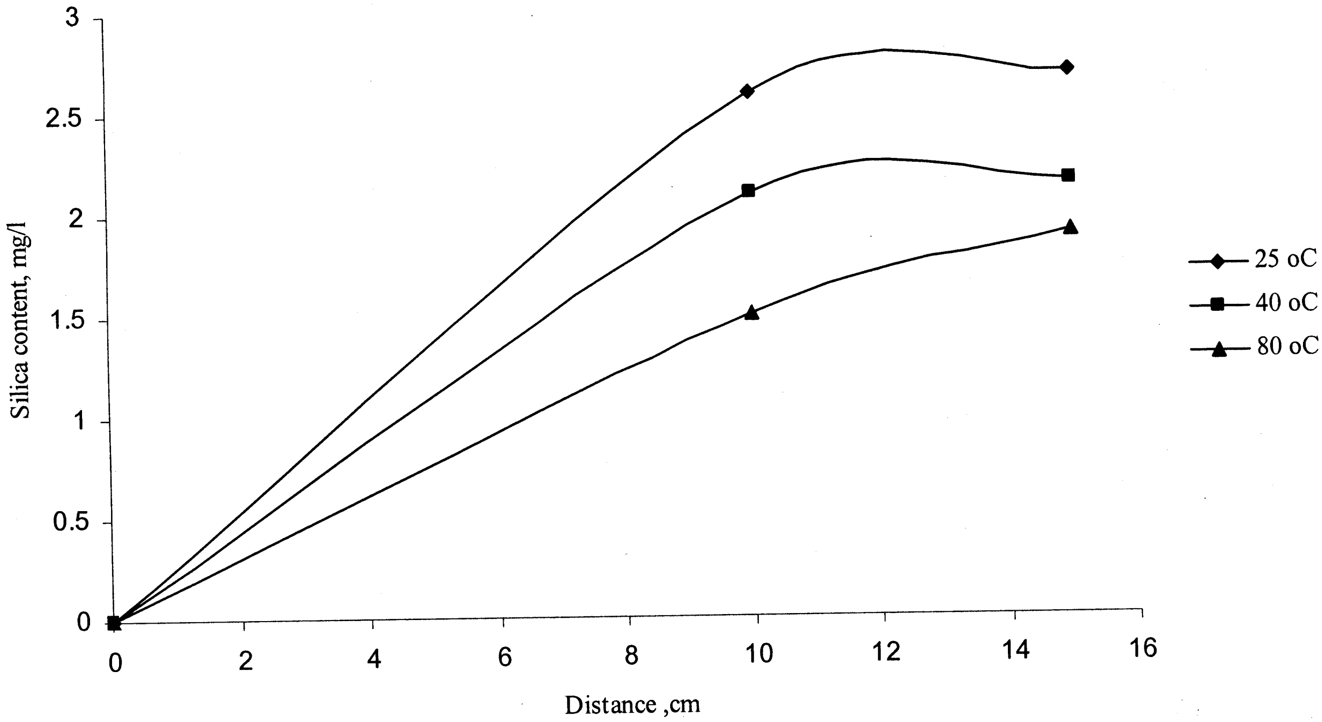

Fig.1— Distance vs silica content in sedimentation (time-30min, flocculant dose-3%)

desilication process. This study presents filtration characteristics of carbonated (desilicated) agro-based BL.

Experimental Setup

BL was procured from local agro-based Paper Mill operating on bagasse, rice straw and wheat straw. At room temperature, BL has following characteristics: initial pH, 13.8; final pH (after carbonation), 10; silica, 2.84; lignin, 30; and RAA (as NaOH), 2.1 g/l at initial pH and 0.3 at final pH. CO_2 is directly passed through BL to bring down pH and also to precipitate silica.

Sedimentation

Batch sedimentation of BL (1 l) was taken in a 28 cm high measuring jar. Different concentrations of flocculent (v/v of BL), Setlyte NPP, from Kaushik Aromatics Ltd, Nagpur were added to the liquor. Samples, which were removed at 5 cm, 10 cm, and 15 cm down the jar at regular interval, were filtered by Whatmann filter paper applying suction. Before filtering, sample was made alkaline by adding dilute NaOH, thereby making the precipitated silica to go back into the solution. The filtrate was analyzed with spectrophotometer using ammonium molybdate method to give the silica content.

Carbonated BL was kept undisturbed and samples were taken at regular interval from locations at 5 cm, 10 cm, 15 cm and 20 cm below the liquid surface in the settling tank and analyzed for silica content using spectrophotometeric method. The experiments were carried out at 25°C, 40°C and 60°C to study the effect on settling rate.

Filtration

Leaf filter (surface area, 500cm^2) was used. Carbonated BL at higher temperature was pumped and time to collect 500 ml filtrate was measured. Rate of filtration at various temperatures were noted under the constant pressure conditions. The results were also noted for various temperatures under the constant pressure conditions with the use of flocculating agent.

Results and Discussion

Sedimentation

Silica settled after 30 min. At room temperature, there was maximum silica at all the locations (Fig. 1). At all the distances down the jar, the silica content at 25°C was higher. Similar results were obtained when the observations were taken after 60 min (Fig. 2). The rate of settling (Fig. 3) recorded at 15 cm below the liquid level using flocculent (3%) shows that the slope of the line is linearly increasing till first 50-60 min and then it becomes sluggish and after some time practically horizontal clearly reducing the settling rate to almost zero. At 25°C, the initial rate of settling is higher with flocculent (3%). The settling rate of silica in carbonated BL with flocculent (5 and 7%) is higher at 40°C (Fig. 3).

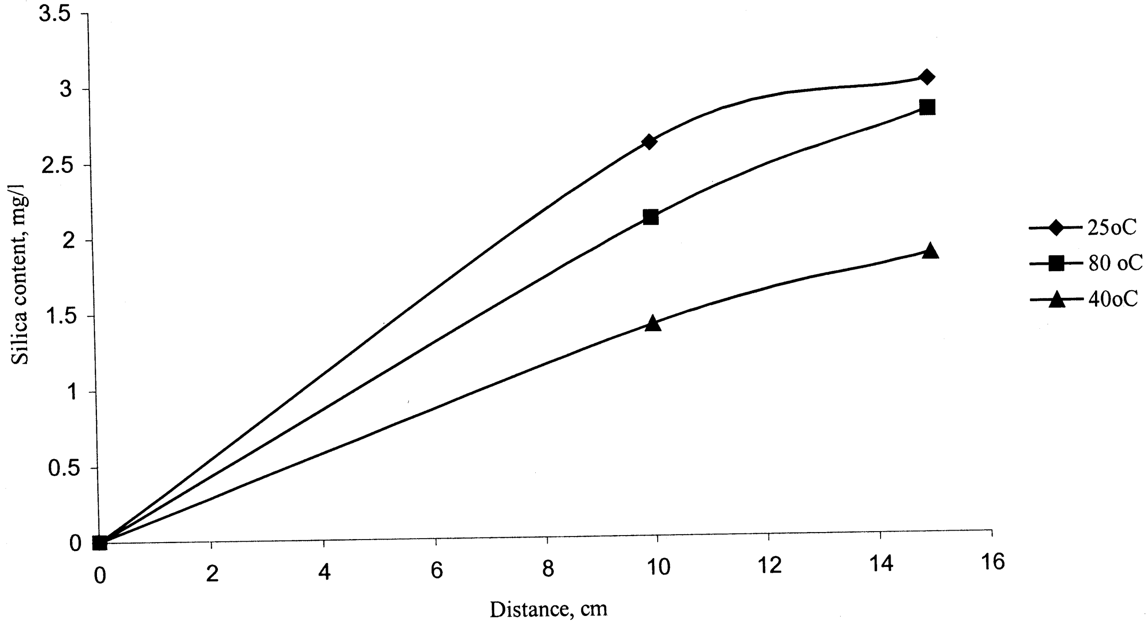

Fig. 2— Distance vs silica content in sedimentation (time-60min, flocculant dose-3%)

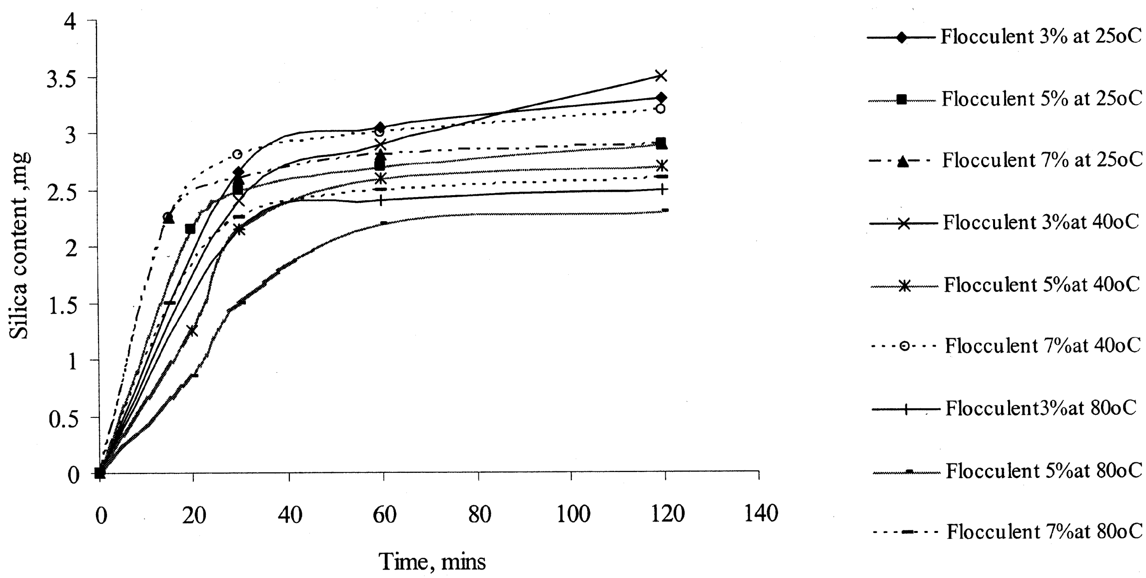

Fig 3— Silica content vs time at different temperature and flocculent dosage (distance-15 cm)

Maximum rate of settling (Fig. 3) is achieved at room temperature with flocculent (3%). The rate of settling with higher flocculent concentration is considerably lower at room temperature. It is observed that the settling is favored by lower temperature and lower dosage. At higher temperature, particles are in excited state of energy and the molecules are in zig-zag motion and hinder the free settling of particles. Silica particle size increases with temperature[3]. The study suggests that the desilication should be carried at higher temperature (Fig. 3) whereas the sedimentation should be done at lower and preferably at room temperature.

Filtration

The filtration characteristics of carbonated BL is strongly influenced by lignin co-precipitation, which is in close proximity that of desilication. Particle size of precipitated silica and its tendency to form aggregates influence the filtration characteristics. Studies were made to find out the influence of temperature on the formation of particle size of SiO_2

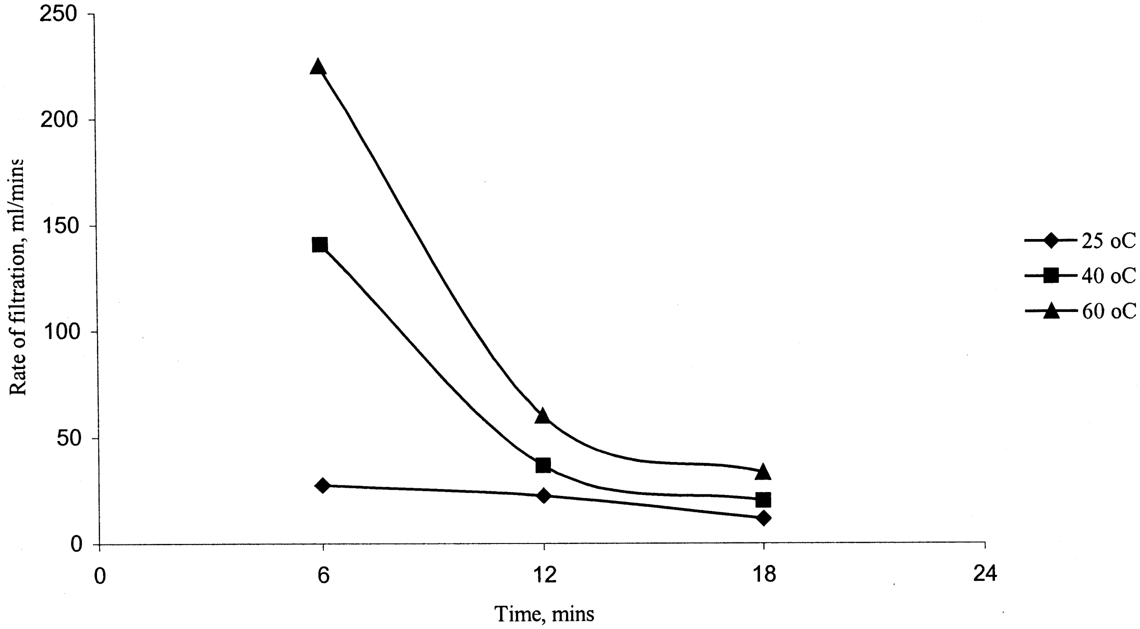

Fig 4— Rate of filtration vs time at different temperature

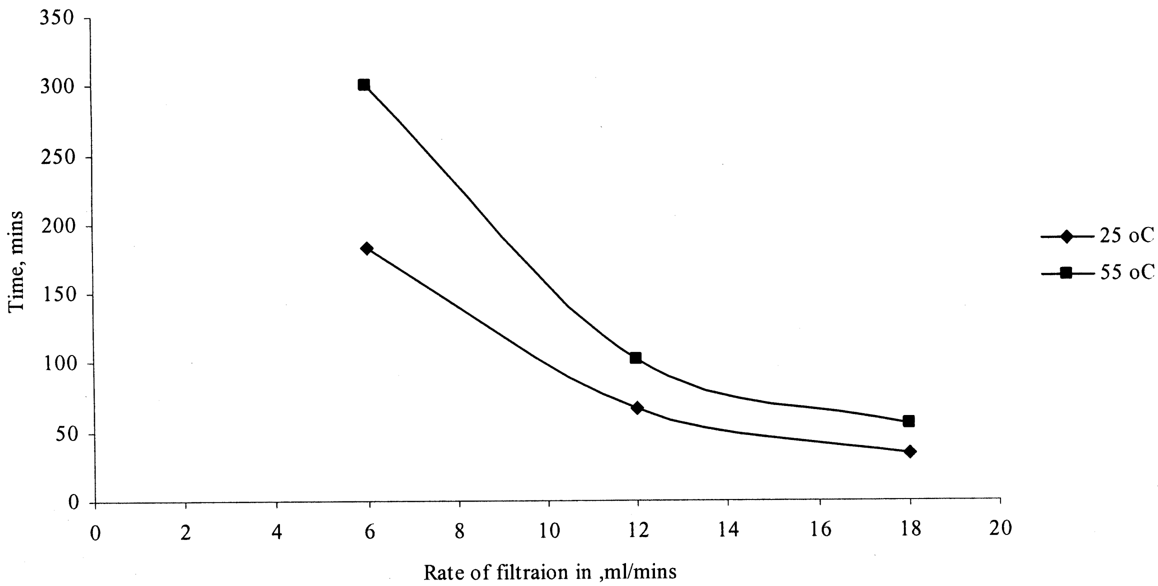

Fig. 5— Rate of filtration vs time at different temperature using flocculent

Average particle size of precipitated silica (1 nm) increases (8 nm) by increasing the temperature to 100°C[3].

Viscosity of agricultural residues-based BL is strongly temperature dependant, more than of wood liquors. The equation for viscosity-temperature relationship formulated[16] is valid for a certain type of BL. Fricke[17] reported the phenomenon of BL viscosity reduction by heat treatment. Kulkarni[18,19] found the reduction of viscosity as a result of breaking up the large molecular mass of lignin polysaccharides complexes.

At 25°C, the rate of filtration is very less and does not change with time (Fig. 4). At 40°C, the filtration rate curve observes downward exponential nature. At 60°C, the initial rate filtration is high and as filtration progresses the precipitated silica and co precipitated lignin deposits on filter medium and offers resistance. This is a resistance in series to filter medium. The thickness of solid deposits on the filter medium increases and progressively the rate of filtration reduce.

It was observed (Figs 5 and 6) that even with flocculent the filtration favors temperature. Rate of

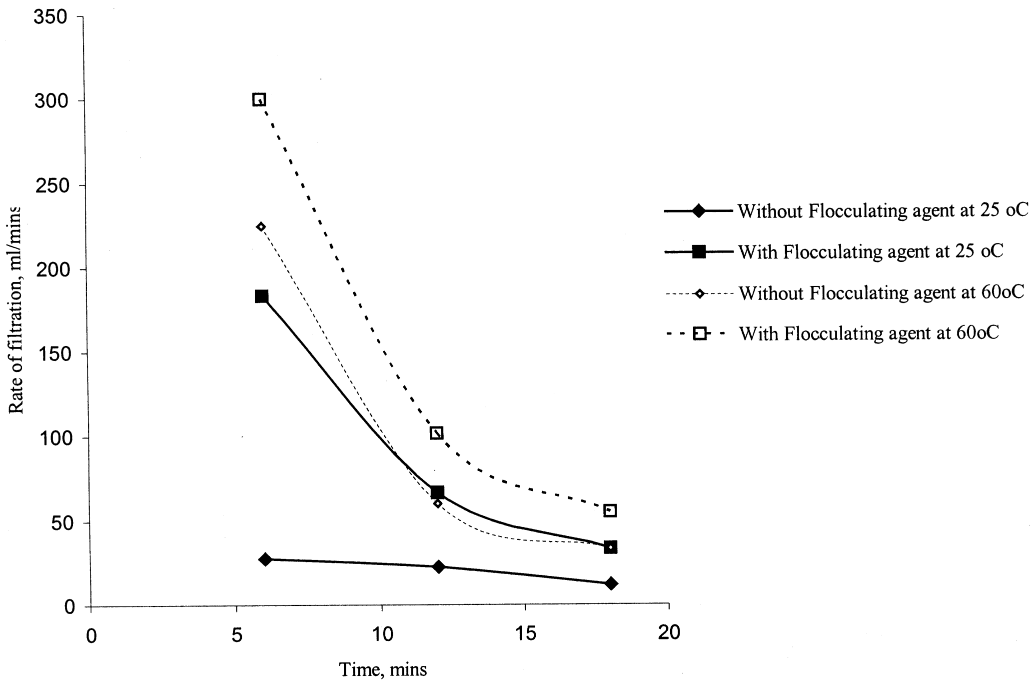

Fig. 6— Rate of filtration vs time at room temperature

filtration decreased with increasing time. The rate of filtration increased with increasing temperature. The flocculating agent was found to give significant increase in filtration rate.

Conclusions

The rate of settling is favored by lower temperature whereas filtration under constant pressure by higher temperature. Flocculent dosage influences rate of settling as well as filtration. Although it is possible to control pH precisely during carbonation, but whenever there is lignin co-precipitation due to storage or slight decrease in pH, the filtration is difficult.

References

1 Kulyan Sundram K & Ganesh R, Non-wood Plant Fiber Pulping Progress Report, *TAPPI*, **3** (1972) 89.
2 Bertel M, A novel desilication technology could prove a valuable boost for straw pulp mills, *Pulp and Pap Asia*, **10** (2000) 107.
3 Mishra D K, Selective removal of silica from alkaline spent liquors, *TAPPI pulping Conf Proc*, 1982, 225.
4 Veeramani H, Significance of silica during pulping: Chemical recovery in bamboo kraft paper mills, in Non Wood Plant Fiber Pulping, Progress Report No. 8, *TAPPI*, 1979, 43.
5 El Ebiary M A, Desilication of black liquor, in Non Wood Plant Fiber Pulping, Progress Report No. 13, *TAPPI*, 1982, 69.
6 Ibrahim H, RAKTA after the modifications, in Non Wood Plant Fiber Pulping, Progress Report No. 19, *TAPPI*, 1991, 139.
7 Chemical recovery plant for small pulp mills using non-wood raw materials, *Terminal Report Project DP/IND/85/048*, UNIDO/CPPRI, 1991.
8 *Proc Int Semin Workshop Desilicat*, UNIDO-SIDA-CPPRI (Hindustan Newprint Ltd., Cochin) 1989.
9 Guimond, Removal of silica, *ABIPC*, **31** (1960) 1162.
10 Iler R K, *Chemistry of Silica* (John Wiley Interscience, New York) 1979, 53-89.
11 Passinen B K, Lignin recovery from black liquor, *Proc Symp Recovery of Chemicals*, Helsinik, 1968.
12 Kopfmann K & Hudeczek W, Desilication of black liquor: Pilot plant tests, *TAPPI J*, **71** (1988) 140-177.
13 Chemical recovery plant for small pulp mills using non-wood raw materials, *UNIDO/CPPRI Terminal Report Project DP/IND/85/048*, December 1991.
14 Whalen M D, A simple method for precipitating easily filterable acid lignin from kraft black liquor, *TAPPI J*, **58** (1975) 110-112.
15 Basu S, Causticization and sludge sedimentation characteristics modifies green liquor, *TAPPI J*, **57** (1974) 101-104.
16 Fellegi J & Shou-zu Hu, Evaluation of technologies on recovery of black liquor chemicals for small and medium size pulp mill using non-wood fibrous raw materials', *UNIDO Report*, 1996.
17 Fricke A L, Black liquor properties, *TAPPI*, 1989.
18 Kulkarni A G, Private communication, *CPPRI*, Saharanpur, India.
19 Kulkarni A G, Pant R & Panda A, Treatment of bagasse black liquor, *IPPTA*, **5** (1993) 4-10.