

# Desilication of Sulphate Weak Black Liquor by the Addition of Lime

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## SUMMARY

The weak black liquor was investigated for removal of silica in it by the addition of slaked lime. Lime, 100-300%, of the theoretical amount of the silica content in the weak black liquor was added in order to find optimum silica removal. It is found that silica, 73-90%, can be removed by the addition of 200-300% lime on silica for 30 minutes at 90°C temperature. It is also found that the lower temperature affects the efficiency of silica removal and optimum temperature seems to be 90°C. Calcium silicate precipitated during desilication process is easily separated from the weak black liquor by filtration in the laboratory study. A pilot plant study to remove silica in weak black liquor by the addition of lime is being studied to know the difficulty in filtration of calcium silicate and loss of organic matter.

## INTRODUCTION

In India conventional raw-material for pulp and paper manufacture is bamboo which contains high percentage of silica (0.5-2%) compared to European woods<sup>1</sup> (0.00-0.007% Silica). Other cellulosic raw material used, such as bagasse (1-1.5% Silica), Straws (4-12% Silica), also contain relatively high concentration of silica compared to woods<sup>2</sup> (0.1-0.3% Silica).

Silica dissolves in the alkaline liquor during sulphate pulping process and forms a soluble sodium silicate in the black liquor. This sodium silicate causes many difficulties during chemical recovery, such as deposition of hard scale on the inside evaporating tubes and pans and coating walls of the recovery boiler causing heat and alkali loss, 3-4 and at the same time affects causticizing efficiency. Lime recovery by reburning lime sludge is not found possible in our country due to high silica content (10%) of lime sludge compared to foreign countries (0.5-1% Silica).

With the view of reburning the lime sludge, laboratory experiments were undertaken to remove silica from weak black liquor by lime addition.

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## EXPERIMENTAL

Weak black liquor (WBL) obtained from sulphate pulping of 20% mixed hardwoods and 80% bamboo (*Dendrocalamus strictus*) was used in the present study. WBL and lime used were first analysed following standard procedures. The analysis results are recorded in Table-1 and 2 respectively for WBL and lime.

TABLE - 1 ANALYSIS OF WEAK BLACK LIQUOR

| S. No. | Analysed for                     | Results |
|--------|----------------------------------|---------|
| 1.     | TW addell at 60°C, °TW           | 13.50   |
| 2.     | T.T.A. as Na <sub>2</sub> O, gpl | 34.10   |
| 3.     | Total solids, W/W %              | 12.35   |
| 4.     | Inorganic content, %             | 37.86   |
| 5.     | Organic content, %               | 62.14   |
| 6.     | Silica content, gpl              | 6.48    |

TABLE - 2 ANALYSIS OF LIME

| S. No. | Analysed for         | Results |
|--------|----------------------|---------|
| 1.     | CaO content, %       | 76.20   |
| 2.     | MgO content, %       | 1.20    |
| 3.     | Inorganic content, % | 95.00   |
| 4.     | Organic content, %   | 5.00    |
| 5.     | Silica content, %    | 5.40    |

## PROCEDURE OF DESILICATION

WBL in an Erlenmeyer flask was kept on hot plate and temperature of the reaction mixture was maintained by the help of energy regulator connected in the line. Calcium oxide (Slaked) 100, 200 and 300% of the theoretical amount against the silica content was added in WBL at 80, 85, 90 and 103°C (B.P. of WBL). The time of reaction was kept constant 30 minutes, in all the experiments except at 90°C temperature it was varied from 40 to 90 minutes, just to see the relationship between reaction time and silica removal (Table-7). Volume of the WBL was maintained time to time, during the experiment by adding distilled water into the reaction flask.

After the experiment, the reaction mixture was filtered through Whatman no.41 (Ashless) filter paper. The residue was washed with hot water and the washings were combined with the above filtrate. Residual silica in the resultant WBL was found out by the help of calorimeter, directly after dilution. Results are recorded in Table-3-7 and also depicted in Figure 1 and 2.

TABLE-3 EFFECT OF ADDED CaO ON SiO<sub>2</sub> REMOVAL (REACTION TEMP. 80°C)

| S. No. | Particulars                          | SiO <sub>2</sub> : CaO |       |       |
|--------|--------------------------------------|------------------------|-------|-------|
|        |                                      | 1 : 1                  | 1 : 2 | 1 : 3 |
| 1.     | WBL Taken L                          | 1.0                    | 1.0   | 1.0   |
| 2.     | CaO Added, g                         | 5.99                   | 11.98 | 17.97 |
| 3.     | Time of Reaction, mts                | 30                     | 30    | 30    |
| 4.     | Initial SiO <sub>2</sub> of WBL, gpl | 6.48                   | 6.48  | 6.48  |
| 5.     | Final SiO <sub>2</sub> of WBL, gpl   | 4.70                   | 2.05  | 1.26  |
| 6.     | SiO removal, %                       | 27.47                  | 68.36 | 80.56 |

TABLE 4 EFFECT OF ADDED CaO ON SiO<sub>2</sub> REMOVAL (REACTION TEMP. 85°C)

| S. No. | Particulars                        | SiO <sub>2</sub> : CaO |       |       |
|--------|------------------------------------|------------------------|-------|-------|
|        |                                    | 1 : 1                  | 1 : 2 | 1 : 3 |
| 1.     | WBL Taken, L                       | 1.0                    | 1.0   | 1.0   |
| 2.     | CaO added, g                       | 5.99                   | 11.98 | 17.97 |
| 3.     | Time of Reactions, mts             | 30                     | 30    | 30    |
| 4.     | Initial SiO of WBL, gpl            | 6.48                   | 6.48  | 6.48  |
| 5.     | Final SiO <sub>2</sub> of WBL, gpl | 4.50                   | 1.94  | 1.08  |
| 6.     | SiO <sub>2</sub> Removal, %        | 30.56                  | 70.06 | 83.33 |

TABLE—5A EFFECT OF ADDED CaO ON SiO<sub>2</sub> REMOVAL (REACTION TEMP. 90°C)

| S. No. | Particulars                          | SiO <sub>2</sub> : CaO |       |       |
|--------|--------------------------------------|------------------------|-------|-------|
|        |                                      | 1 : 1                  | 1 : 2 | 1 : 3 |
| 1.     | WBL Taken, L                         | 1.0                    | 1.0   | 1.0   |
| 2.     | CaO Added, g                         | 5.99                   | 11.98 | 17.97 |
| 3.     | Time of Reaction, mts.               | 30                     | 30    | 30    |
| 4.     | Initial SiO <sub>2</sub> of WBL, gpl | 6.48                   | 6.48  | 6.48  |
| 5.     | Final SiO <sub>2</sub> of WBL, gpl   | 4.28                   | 1.75  | 0.66  |
| 6.     | SiO <sub>2</sub> Removal, %          | 33.95                  | 72.99 | 89.81 |

TABLE—5B EFFECT OF ADDED CAO ON SiO<sub>2</sub> REMOVAL (REACTION TEMP. 90°C)

| S. No. | Particulars                          | SiO <sub>2</sub> : CaO |       |       |
|--------|--------------------------------------|------------------------|-------|-------|
|        |                                      | 1 : 1                  | 1 : 2 | 1 : 3 |
| 1.     | WBL Taken, L                         | 1.0                    | 1.0   | 1.0   |
| 2.     | CaO Added, g                         | 2.99+                  | 5.99+ | 8.98+ |
|        |                                      | 2.99                   | 5.99  | 8.98  |
| 3.     | Time of Reaction, gpl                | 15+15                  | 15+15 | 15+15 |
| 4.     | Initial SiO <sub>2</sub> of WBL, gpl | 6.48                   | 6.48  | 6.48  |
| 5.     | Final SiO <sub>2</sub> of WBL, gpl   | 4.25                   | 1.62  | 0.64  |
| 6.     | SiO <sub>2</sub> Removal, %          | 34.41                  | 75.00 | 90.12 |

TABLE—6 EFFECT OF ADDED CaO ON SiO<sub>2</sub> REMOVAL (REACTION TEMP. 103°C)

| S. No. | Particulars                          | SiO <sub>2</sub> : CaO |       |       |
|--------|--------------------------------------|------------------------|-------|-------|
|        |                                      | 1 : 1                  | 1 : 2 | 1 : 3 |
| 1.     | WBL Taken, L                         | 1.0                    | 1.0   | 1.0   |
| 2.     | CaO, Added, g                        | 5.99                   | 11.98 | 17.97 |
| 3.     | Time of Reaction, mts                | 30                     | 30    | 30    |
| 4.     | Initial SiO <sub>2</sub> of WBL, gpl | 6.48                   | 6.48  | 6.48  |
| 5.     | Final SiO <sub>2</sub> of WBL, gpl   | 4.16                   | 1.58  | 0.56  |
| 6.     | SiO <sub>2</sub> removal, %          | 35.80                  | 75.62 | 91.36 |

TABLE-7 RELATION BETWEEN TIME OF REACTION AND  $\text{SiO}_2$  REMOVAL (REACTION TEMP.  $90^\circ\text{C}$ )  
Sample—IL, WBL OF 6.48  $\text{SiO}_2$  gpl.

| S. No. | $\text{SiO}_2 : \text{CaO}$ | CaO Added (g) | Time of Reaction (Min.) | Final $\text{SiO}_2$ in WBL (gpl) | $\text{SiO}_2$ Removal (%) |
|--------|-----------------------------|---------------|-------------------------|-----------------------------------|----------------------------|
| 1.     | 1:1                         | 5.99          | 30                      | 4.28                              | 33.95                      |
| 2.     | 1:1                         | 5.99          | 60                      | 3.41                              | 47.37                      |
| 3.     | 1:1                         | 5.99          | 90                      | 2.10                              | 67.59                      |
| 4.     | 1:2                         | 11.96         | 30                      | 1.75                              | 72.99                      |
| 5.     | 1:2                         | 11.93         | 60                      | 1.28                              | 80.24                      |
| 6.     | 1:2                         | 11.98         | 90                      | 0.65                              | 89.99                      |
| 7.     | 1:3                         | 17.97         | 30                      | 0.66                              | 89.81                      |
| 8.     | 1:3                         | 17.97         | 60                      | 0.54                              | 91.66                      |
| 9.     | 1:3                         | 17.97         | 90                      | 0.42                              | 93.51                      |

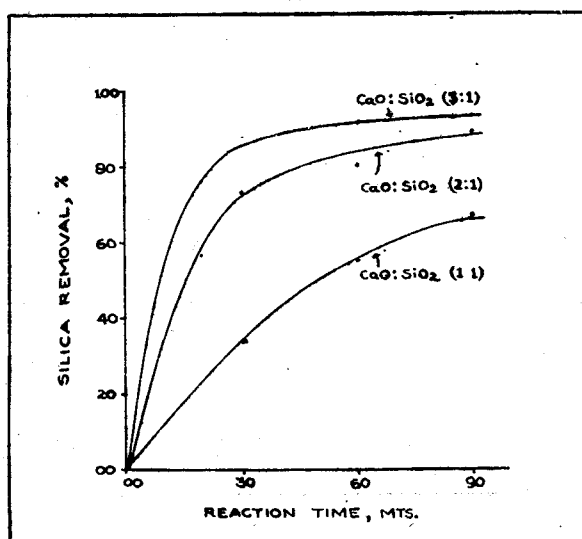
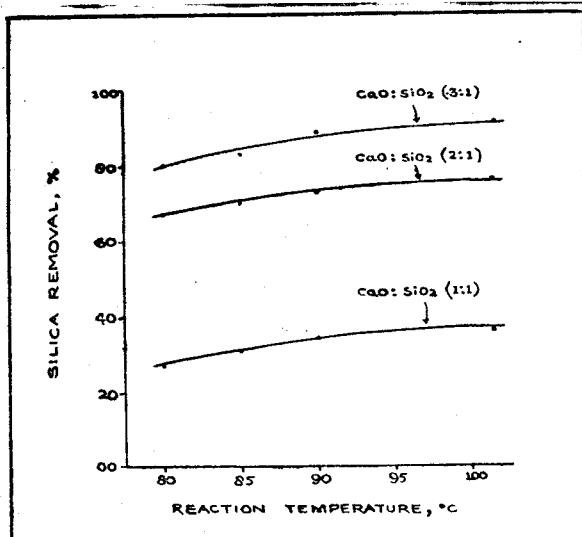


Fig. 2

## RESULTS & DISCUSSION

Desilication of WBL by the addition of lime is very useful especially when higher percentage of bamboo, bagasse or straws are used as a fibrous raw-material for pulp manufacture in order to eliminate various difficulties encountered during recovery process mentioned earlier and return the lime sludge.

It is found that 73-90% of silica can easily be removed from WBL by the addition of 200-300% lime on silica content at  $90^\circ\text{C}$  for 30 minutes.

Calcium silicate, precipitated by the addition of lime is easily filterable from the WBL in laboratory investigation.

It is also found that the lower temperature affects the efficiency of silica removal. The optimum temperature seems to be  $90^\circ\text{C}$ .

Rate of desilication is greatly effected by the lime of reaction especially when  $\text{CaO} : \text{SiO}_2$  ratio is high (Figure-2)

Mode of addition of lime does not seem to affect the efficiency of silica removal of WBL as silica removal percentage is almost equal in both the cases when lime was added at a time (Table 5A) or in parts (Table 5B).

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