Conversion of Black Liquor From Agricultural Residues Into Sodium Carbonate Pellets in Fluidised Bed Reactor At Shreyans Papers

Sharma N.K., Bhargava K.S. and Sharma S.K.

ABSTRACT

Black liquor from agricultural residues having high silica content is being converted into sodium carbonate pellets in fluidised Bed reactor (FBR) at Shreyans Papers. To avoid hard scale of silica deposits in Multiple effect Evaporator tubes black liquor is concentrated only to 25% total solids in evaporators, pH of black liquor in all stages i.e. WBL, SBL and HBL is being maintained around 11.0 to avoid Silica and Lignin precipitation and to have better fluidity of black liquor. Chlorides and Potassium content in black liquor are kept low to maintain fluidisation of Sodium carbonate pellets bed in the reactor. Temperature control at various stages is the key for running Fluidised Bed Reactor successfully and economically.

Constant check is required to maintain the required particle size distribution of sodium carbonate pellets in FBR bed.

These are salient features by which Shreyans Papers has overcome initial teething troubles to convert black liquor into Sodium Carbonate pellets successfully.

Key words

Bed height, fluidisation, defluidisation, wind box. temperature, upper bed temperature, lower bed temperature, atomising air, fluidising air, airpreheater, product silo, pellets, pellets dissolving, temperature, Thermo Couples, Superficial Space Velocity, product cooler, venturi scrubber, Gritt cyclone.

INTRODUCTION

Continuously plagued by ever increasing shortage of conventional raw materials like soft woods, hard wood and Bamboo, India started looking towards agro based cellulosic raw materials like Bagasse, Wheat and Rice straw and various grasses to meet ever increasing demand of paper. As more and more of agro based raw materials were used, the need for having a viable "Soda Recovery System from agro based raw materials has become necessary. High viscosity and high silica contents from agro based raw materials make it very difficult to

Shreyans Industries Ltd.
(Unit: Shreyans Papers)
P.O. Ahmedgarh-148 021
Distt. Sangrur (Pb.)

go in for conventional Soda Recovery system where very high concentration of black liquor (60%-65%) is required for incineration. In absence of any alternate viable recovery system agro based paper mills became a major source of heavy pollution because of discharging their effluent without recovery of caustic soda. It is in this context that search for a proper recovery system suitable for non conventional raw materials became big need of paper industry. Shreyans Paper located in Ahmedgarh, Sangrur (Punjab) and based on agro raw materials like Bagasse, Wheat and Rice straws etc and having 80 TPD paper production capacity made a break through and offered to become pioneer to take a big challenge in installing a non-conventional Soda Recovery System based on Fluidised Bed Reactor. With such process technology plant is tried for the first time in India. Such plants are in operation in South Africa, Mexico and USA. Soda Recovery system based on fluidisation bed reactor to burn black liquor with 45% solids is in operation and is running successfully for the last two months at Shreyans Papers paving the way for installing similar plant in other paper units using agro based raw materials for manufacture of paper.

SALIENT FEATURES

The Soda Recovery plant at Shreyans papers mainly consist of:

1. Sextuple Effect Evaporator for 600 tons water evaporation per day consisting of one heat exchanger with forced circulation system.

2. Fluidised Bed Reactor for burning 75 tons solids per day having DCs controls.

3. A recausticizing plant for the production of 20 tons caustic soda per day as white liquor having strength 100 gms per litre.

BRIEF DESCRIPTION OF THE PROCESS:

Multiple effect evaporator Weak black liquor is fed as split feed to no.4 and 5 effect, where as live steam is fed in to heat exchanger weak liquor travels forward from no.6 effect with the help of transfer pumps. Vapour travels from Ist effect vapour separator to no.6 effect where a vacuum of 660 mm of mercury is maintained with the help of vacuum pump. Fresh condensate is removed from Heat exchanger to condensate tank to be pumped to DM water tank. Combined condensate is removed from no.6 effect and is used in the process. The semi concentrated black liquor of 20 to 25% solids is discharged from the evaporator to semi concentrated black liquor tank. **(Fig.I)**.

FLUIDISED BED REACTOR

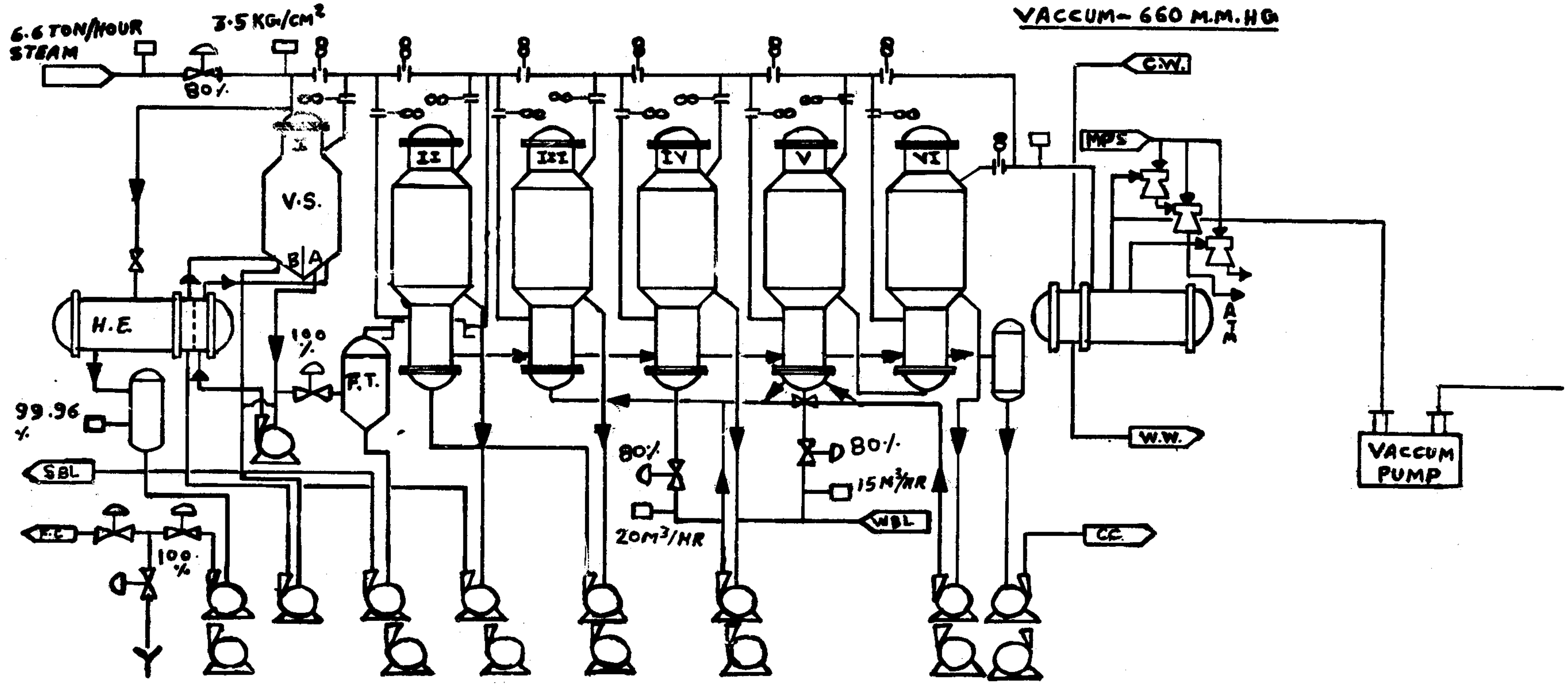
Semi concentrated Black liquor is further concentrated to 40 to 45% solids by circulating in Venturi cycline system of FBR with the help of flue gases coming out from FBR.

This concentrated black liquor is termed as Heavy black Liquor (HBL) and is stored in HBL tanks and is fired in the reactor to generate Sodium Carbonate Pellets as final product, which can be either stored in Product Silo or to be dissolved in Product dissolving tank to produce Green liquor (Figure II) Secondary Scrubber having weak wash liquor/water as scrubbing medium has been provided to have suspended particulate matters below 100 mg/Nm^3 in the flue gases discharged to atmosphere. **(Figure 2).**

A number of hurdles and teething problems have been faced in the initial stages requiring a number of modification and changes to run the Fluidised Bed Reactor without any interruption and defluidisation of the bed. FBR technology is perhaps a key to successful conversion of black liquor having high Silica content from agricultural residues to Sodium Carbonate pellets. Sodium Carbonate pellets are dissolved in Weak wash from Recausticizer plant to make Green liquor and Green liquor is converted into white liquor by treating with lime in conventional Recausticizer plant. The experiences in operating a fluidised bed reactor are described in the following paragraphs.

A. MANPOWER PLANNING

1. Since skill and semi skill persons for fluidised bed reactor were not available. Fresh trainees at all levels were recruited before erection of the plant.



F.T. → FLASH TANK

H.E. → HEAT EXCHANGER

V.S. → VAPOUR SEPARATOR

L.P.S. → LOW PRESSURE STEAM (3.5 KG/CM²)

M.P.S. → MEDIUM PRESSURE STEAM (10 KG/CM²)

F.C. → FRESH CONDENSATE

C.C. → COMBINED CONDENSATE

C.W. → COOLING WATER

W.W → WARM WATER TO COOLING TOWER

FEED WEAK BLACK LIQUOR 8% SOLIDS

OUT SEMI CONCENTRATE BLACK LIQUOR 20 TO 25% SOLIDS

MULTIPLE EFFECT EVAPORATORS

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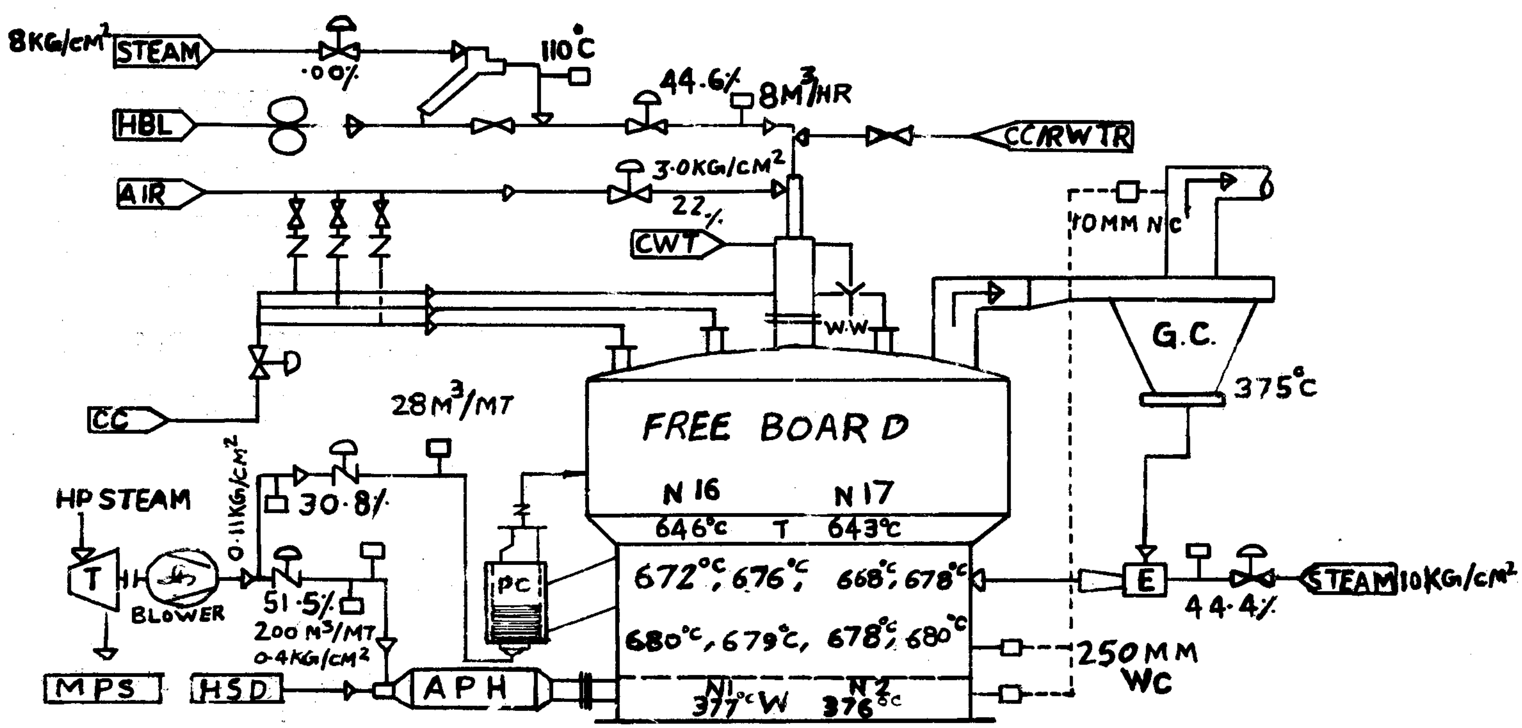
Fig.1

FLUIDISED BED REACTOR TEMPERTURE FLOW AND PRESSURE AT VARIOUS ZONES



T → TURBINE

HP → HIGH PRESSUR STEAM 42KG/CM²

MP → MEDIUM PRESSUR STEAM 8 TO 10 KG/CM²

FA → FLUIDISING AIR

APH → AIR PREHEATER

W → WIND BOX

LB → LOWER BED

U.B. → UPPER BED

WW → WARM WATER

PC → PROUCT COOLER

S → SODIUM CARBONATE PELLETS

G.C → GRIT CYCLONE

E → EDUCTOR

CC → COMBINED CONDENSATE

HBL → HEAVY BLACK LIQUOR 40–45% SOLIDS

T → TRANSITION ZONE

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2. They were given education on Fluidised bed reactor and were associated with equipment/ instrument installation at the site.

3. The Graduate trainees were given training on DCS, field and control instruments at supplier workshop.

4. Due to high level of instrumentation only two operators and three helpers per shift are required to run both M.E. evaporator and Fluidised Bed Reactor.

5. During the commissioning of Fluidised Bed Reactor, a person experienced in operation of FBR and one assistant were kept for few days to help in commissioning of FBR.

B. FLUIDISED BED SYSTEM OPERATION

The following procedure is being followed to start FBR

1. Secondary and primary venturi scrubber sumps are filled upto a desired level with water and semi concentrated black liquor respectively and liquors are recirculated. Temp. of feed gun jacket is kept less than 50°C by circulating cold water through the jacket of feed gun. A 500 BHP blower is started and purging and atomising air system are activated and fluidization air is passed through air preheter.

Atomising air is passed through feed gun continuously. Then HSD firing is started to raise temperature in wind box to 540°C at the rate of 40°C per hour. The temperature is slowly raised as to protect the life of refractory provided in side air, preheater, Fluidised Reactor main body, ducting and Grit cyclone.

Sodium carbonate pellets are used as medium for fluidisation. Sodium carbonate pellets were introduced in the bed when wind box temperature of 540°C is attained at a rate of 200-250 mm of bed height per hour. A minimum superficial space velocity of 67 cm/sec of air is to be maintained throughout the bed.

After attaining the desired bed height 183 cms Charcoal injection is started to raise temperature of the bed to 680°C at the rate of 80°C per hour.

Black liquor firing is started when the bed temperature reaches 680°C. The quantity of black liquor fired is controlled to ensure that the bed temperature is between 700°C (maximum) and 660°C (minimum) depending upon the characteristics of black liquor.

A bed height of 150 to 210 cm (average 183 cm) is being maintained. Depending upon characteristics of black liquor more bed depth improves operational stability and bed differential pressure, but requires more volume of fluidising air. To avoid dust particles developing stickiness Free board temp. is maintained below 600°C by combined condensate spray through three cooling spray guns.

Discharge of pellets is done through product cooler, which also has fluidisation air and cool water circulation its double jacketed body. Product cooler discharges the material through a rotary valve to metering screw which is also water cooled. The pellets are discharged at 80 to 100°C to either bucket elevator or to pellets dissolving tank. There is provision in the system to store pellets in the product silo through bucket elevator from where Sodium pellets can be discharged to attirition mill or pellet dissolving tank or for introduction to reactor to make the bed through rotary valve, or to be filled in the bags for other chemical industries.

C. OPERATIONAL PROBLEMS AND REMEDIES

For first start-up to have desired pellets size distribution in the fluidised bed reactor imported Sodium Carbonate pellets were used. On investigation, it is found that dense sodium carbonate granules can be used for FBRs bed material. Now currently the product from the unit is being used as bed charging material.

For favourable fluidisation, the size distribution particles in the bed should be as follows:

ASTM Mesh size	Retention %
10	2
20	10-35
30	15-35
40	15-30
50	8-20
60	2-10

CONTROL OF FINES IN THE BED

Close control of particles size and distribution must be kept. If more than 45% particles of same size are present in the bed, there will be more voids, offering less resistance to air passage, resulting in narrower fluidisation band with same air flow. If the percentage of fines goes below 2% there is chance for defludisation of bed as more void space is created by the absence of fines. All cares are taken to have minimum 3.5% fines in the bed. The following methods are being followed to increase the percentage of fines in the bed.

a. Introduction of fines by reducing the size of Sodium carbonate pellets in the attrition mill and feeding the same into the reactor with the help of an eductor.

b. Increasing feed gun pressure.

c. Increasing the fluidisation air flow.

d. Keeping bed temperature low to the extent which does not disturb self combustion of black liquor.

e. Increasing the bed depth for stable operation and increasing bed differential pressure.

f. Increasing feed rate of black liquor in to reactor.

g. Decreasing wind box temperature.

h. Controlling Chloride and Potassium in the black liquor.

S.No.	Raw Material	Chlorides as NaCl%
1.	Wheat straw	0.8 to 1.6
2.	Bagasse	0.1 to 0.2
3.	Sarkanda	1.10
4.	Rice straw	0.40 to 1.4
5.	Jute caddy	0.10

The presence of high Chloride and Potassium in the black liquor the eutectic point of Sodium Carbonate pellets. If the Chlorides content as NaCl in black liquor exceed 0.6% on solid basis, there is sharp decline in the softening point of Sodium Carbonate pellets produced, and the chances of defluidisation of the bed will increase and it may also result in lumps formation in the bed.

The Chloride contents in various raw materials are given below:

S.No.	Temp. of wash water ^0C	Soluble Chlorides in washed wheat straw, as NaCl%
1.	40	0.1
2.	60	0.08
3.	70	0.075
4.	80	0.07

A series of experiments were conducted to reduce the Chloride content in Wheat straw by simple fresh water treatment as the Chlorides present in agricultural residues are highly water soluble.

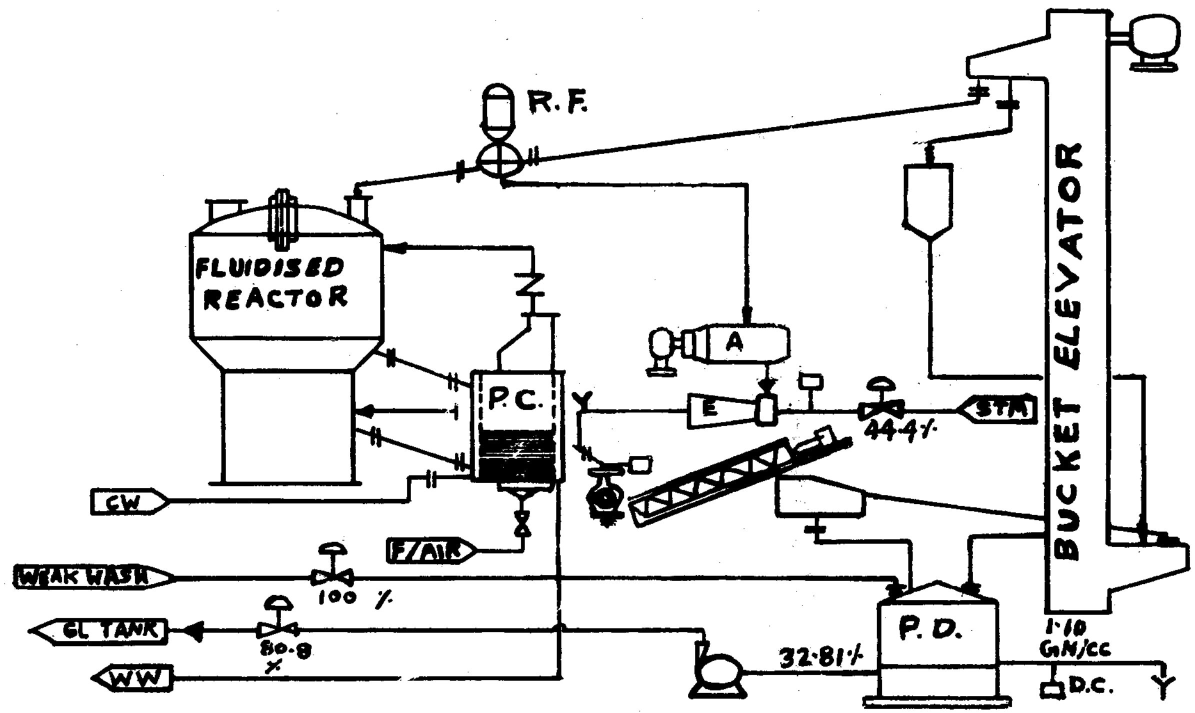
Chloride contents in Wheat straw, after washing the same at 2.5% consistency are tabulated below:

As mentioned above, the Chloride contents as NaCl in natural Wheat straw are found to be arround 1.125% w/w and the mill water has a concentration of 75 ml/ltr. of Chlorides as NaCl. It is essential to remove Chlorides content from agricultural residues before cooking so that black liquor obtained does not have more than o.6% Chlorides as NaCl. It is desirable to use Caustic Soda having low Chloride content to avoid high clorides in black liquor. It is recommended that a suitable Chloride purging unit is provided in the recausticizer plant as to get white liquor of low Chloride content.

BED TEMPERATURE

Bed temperature plays an important role both for agglomeration of Sodium Carbonate pellets in the bed resulting in defluidisation as well as burning of black liquor. By decreasing bed temp. the percentage of fines in the bed can be increased. The reactor has been divided in to various sections, wind box, lower bed, upper bed, Transition zone and Free board. A number of Thermo Couples have been

FLUIDISED BED REACTOR PRODUCT SODIUM CARBONATE HANDLING SYSTEM



RF → ROTARY FEEDER
F/AIR → FLUIDISED AIR
STM → MEDIUM PRESSURE STEAM 8 KG/CM^2
BE → BUCKET ELEVATOR
PD → PELLET DISSOLVING TANK
A → ATTRITION MILL

E → EDUCTOR
PC → PRODUCT COOLER
CW → COOLING WATER
WW → WARM WATER
GL → GREEN LIQUOR
DC → DENSITY CONTROLLER

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Fig.3

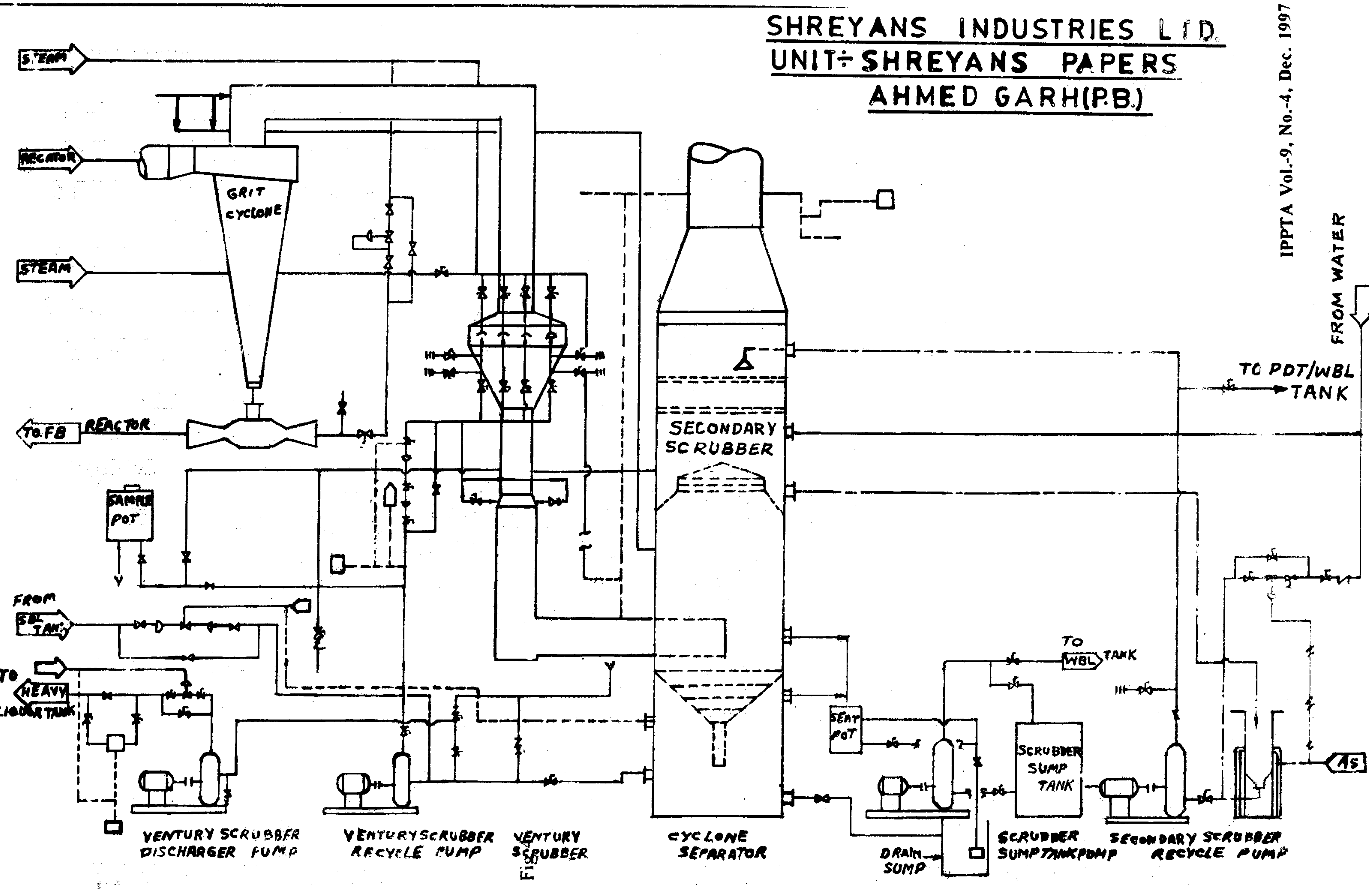

Fig-4

REMOVAL OF SUSPENDED PARTICULATE MATTERS FROM FLUE GASES DISCHARGED FROM FBR provided to monitor the temperature at various sections of the reactor especially at upper and lower bed. 3 nos. of cooling spray guns have been provided to maintain free board temperature of the reactor. High bed temp. may result in softening of Sodium Carbonate pellets hence may cause lump formation in the bed. Low bed temp. results in poor combustion of black liquor and more fines in the system thereby increasing load on Grit Cyclone. High percentage of fines in flue gases also increase percentage inorganic in Heavy Black liquor (HBL) resulting in poor burning of heavy black liquor.

BED DIFFERENTIAL PRESSURE BAND

A constant watch is kept not to allow bed differential pressure band getting narrow as it is an indication of defluidisation. Proper fluidisation of the bed is of utmost importance and is the key to successful operation of fluidised bed reactor. As soon as defluidisation signs are observed the black liquor firing is promptly cut as a first step to avoid any defluidisation. Steam lancing is resorted to obtain refluidisation condition which is indicated in the fluidised band.

The following steps are being taken to avoid defluidisation.

Size distribution of Sodium Carbonate pellets is checked. In case of low fines, and fines are introduced through attrition mill. Bed temperature is brought down to minimum where auto combustion of black liquor takes place. Chlorides and potassium content in black liquor are kept low. The percentage of pellets of one particular size between 20 to 50 ASTM mesh should not exceed 45%.

Fluidizing air flow is maintained as to have minimum superficial space velocity at 0.73 Metre/sec. and free Oxygen in outlet gases around 2.5%.

BLACK LIQUOR FEED GUN POSITION

Black liquor feed gun position depends upon the nozzle size and characteristic of black liquor. In the present case it has been fixed at the height of 550 cms from Orifice plate. A desirable spray pattern is one that covers an area equal to 2/3 rd of the inside diameter of the reactor. No black liquor should drop on the wall of the reactor. The droplet size should be mostly between 2.4 to 4 mm in diameter.

The following operational parameters are recommended to have normal running of fluidised bed reactor based on the commissioning experience.

- Windbox temp.- minimum 200^0
- Fluidising air pressure- 0.39 to 0.41 kg/cm^2
- Differential pressure between orifice plate and bed : 152-183 cms WC
- Differential pressure across windbox and orifice plate: 249-254 cms WC
- Differential pressure between free board and Grit cyclone: 10-20 cms WC
- Differential pressure between outlet of Grit Cyclone and ventgguri scrubber 38-50 cms WC
- Air flow to product cooler: 22.66-28.32 cu m/min
- Temp. of the bed 660-690^{0}C
- Difference in temp. between upper and lower bed 8^{0}C Maximum
- Transition zone temp. 660-676^{0}C
- Free board temp. 580-612^{0}C
- Concentration of heavy black liquor 45% solids

The purity of Sodium carbonate pellets obtained from FBR is found to be around 94%. Green liquor obtained by dissolving Sodium Carbonate pellets is easily converted into white liquor in recausticizer plant and found to be clear.

CONCLUSION

Fluidised Bed Reactor is found to be suitable for converting black liquor having high Silica into Sodium Carbonate pellets. The most critical area in operation is maintaining the fluidisation of bed. This is done by controlling the following:

1. Bed temp. in different zones.

2. Differential pressure at various stages.

3. Maintaining Chloride and Potassium level in black liquor.

4. Maintaining size distribution of Sodium Carbonate pellets.

It is important to have proper field & instruments and DCS to control the operations of fluidised bed reactor which can be easily run by two operator and three helpers.

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