Precipitated Silica from Rice Husk Ash

ABETS

CGPL, IISc

Motivation

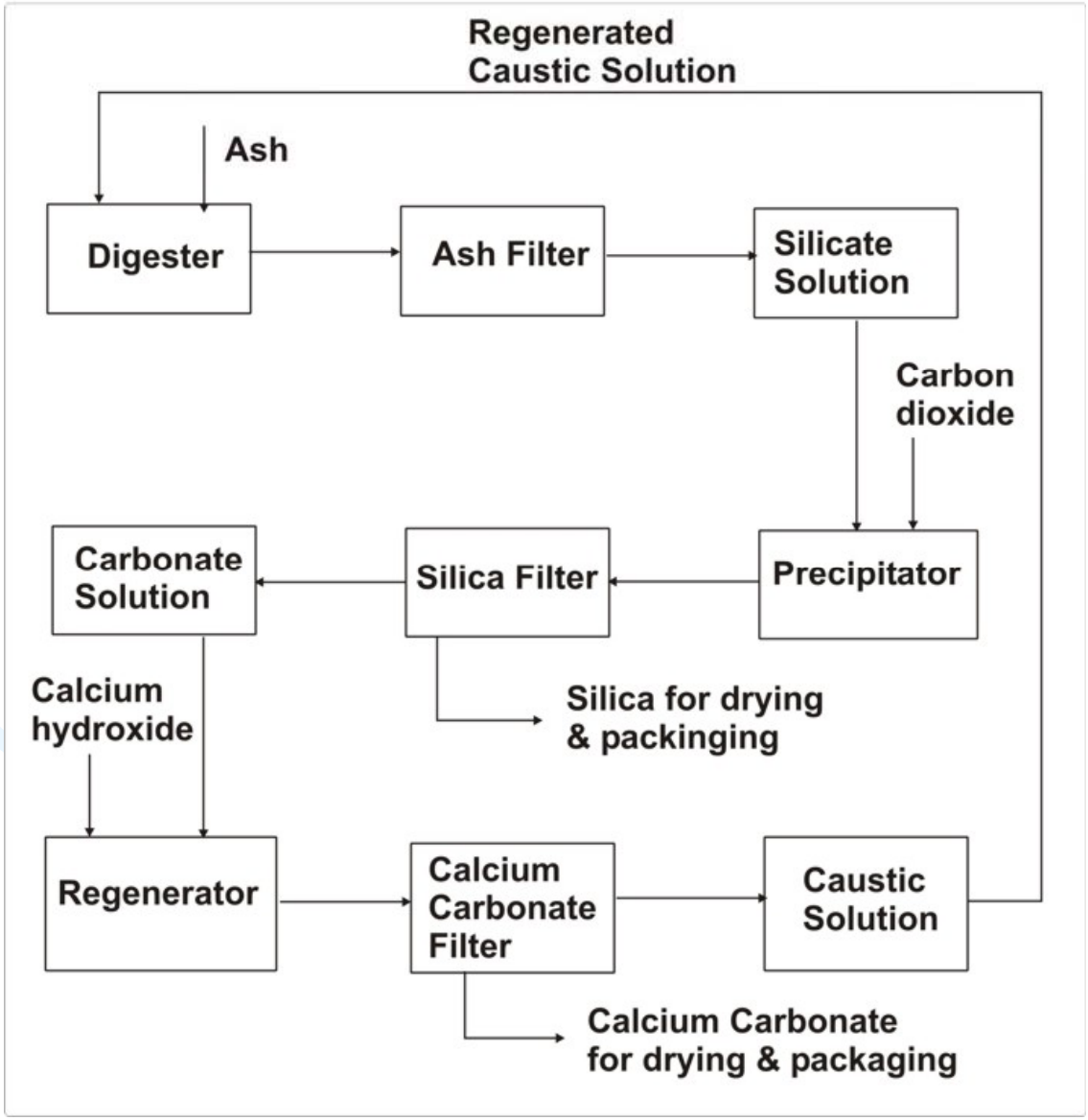
- Rice husk is a widely available agricultural waste India produces around 25 million tons of Rice Husk
- It is largely used as a fuel—in small scale, and in large scale for electrical power generation and thermal needs
- Rice husk contains 20 % ash and leaves large amount of residue (about 25 %) after it is burnt causing a disposal problem
- Silica is the main constituent of the Rice husk ash (~ 90 %)
- Precipitated silica is a high value product (Rs. 40 per kg) having applications in rubber, cosmetics, tooth paste and many other industries
- Production of precipitated silica from rice husk thus solves the disposal problem ash and provides additional revenue stream
- This process is cheaper – production cost about Rs. 22 – 24/kg of silica

Uses of Precipitated Silica

- Rubber industry – as an reinforcing agent
- Cosmetics
- Tooth pastes – as an cleansing agent
- Food industry – as an anti-caking agent.
- Paints – as a filler

The Process

Flow Chart of Precipitated Silica



Digestion

- Heating of rice husk ash with caustic solution at specific conditions results in extraction of insoluble silica from ash as soluble sodium silicate.
- The slurry is filtered and clear filtrate taken for precipitation.
- Residual undigested ash containing mainly carbon with Iodine number of 300 – 400 being tried for improving the same/for water treatment.
- $\chi SiO_2 + 2NaOH \dashrightarrow Na_2O.\chi SiO_2 + H_2O$

Precipitation

- The most crucial step carried out in two stages.
- Carbon dioxide is passed into the sodium silicate solution at designed conditions.
- Sodium silicate reacts with carbon dioxide to form Precipitated Silica and Sodium carbonate.
- The slurry is filtered and clear filtrate taken for re-precipitation for removal of small quantities of unprecipitated silica.
- Reprecipitation carried out similarly to precipitation – slurry is filtered and the clear filtrate taken for Regeneration.
    - $Na_2O.\ XSiO_2 + CO_2 \text{----}\rightarrow XSiO_2 + Na_2CO_3$

Regeneration

- The caustic required for Digestion is regenerated in this step.
- Sodium carbonate solution reacts with calcium hydroxide to form caustic solution and precipitated calcium carbonate.
- The calcium carbonate slurry is filtered and the clear caustic solution is taken for Digestion.
- Precipitated calcium carbonate is marketed/or it can be heated give Calcium oxide and used again for the regeneration.
- 95 % of the caustic used is regenerated.
  - $Na_2CO_3 + Ca(OH)_2 \text{----} \rightarrow 2NaOH + CaCO_3$

Typical Properties of Precipitated Silica

- Nature: Amorphous powder
- Appearance: White fluffy powder
- Purity: $\geq$ 98 %
- Surface Area: 100 – 350 m^2/g
- Bulk density: 100 – 400 g/l
- Loss on Ignition: 3.0 – 6.0 %
- pH of 5 % slurry: 6.3 $\pm$ 0.5
- Particle size: 1–30 µm

Features of the Process

- All operations are carried out at temperature less that 100°C
  - Current commercial production is from sand and uses 1500°C for digestion
- CO_2 is used for precipitation—Caustic can be regenerated. The byproduct, precipitated calcium carbonate is a marketable—or $Ca(OH)_2$ can be regenerated
  - Current commercial production utilizes acid precipitation.
- Environmentally friendly as the chemicals used are regenerated. Rice husk ash a nuisance at large capacities is taken care off.
  - Current commercial production using acid precipitation results in formation of sodium sulphate as effluent and requires effluent treatment.
- Comparatively low cost of production at around Rs. 22 - 24/kg (by around 20 %) compared to commercial process of silica and value addition by sale of calcium carbonate.
- Green Silica – Renewable raw material source compared to conventional process using sand from beautiful beaches

Plant Economics

Estimated Ash requirement for 1 ton of silica: 1.6 tons dry		Silica Capacity – 24 MT/day Plant Cost ~ Rs. 240 million	Silica Capacity – 5.0 MT/day Plant Cost ~ Rs. 65.0 million
Sl. No	**Particulars**	**Cost in Rs. Per kg of Silica**	**Cost in Rs. Per kg of Silica**
1.	Raw Material	7.0	7.0
2.	Cost of Manpower	2.0	2.5
3.	Maintenance Cost	1.6	1.9
4.	Power cost @ Rs. 4.0/kwh	4.1	4.5
5.	Packing, Handling & Transportation	2.0	2.0
6.	Thermal energy	6.1	6.3

Plant Economics - continued

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Sl. No	**Particulars**	**Cost in Rs. Per kg of Silica**	**Cost in Rs. Per kg of Silica**
8.	**Total Production Cost**	**22.80**	**24.20**
9.	Revenue Generated Cost of Silica/kg Cost of CaCO3/kg **Total**	 40.0 6.0 **46.0**	 40.0 6.0 **46.0**
10.	Depreciation (10 years)	3.0	3.6
11.	Interest @ 10 %	3.0	3.6
12.	**Net Profit**	**17.20**	**14.60**

Problems resolved during the Development

- Controlling the properties of the product
  - Purity and other chemical properties were easy to control
  - Surface area and bulk density were much more difficult to control
  - The precipitation step is the critical step
  - More than 100 experiments conducted to identify the controlling parameters

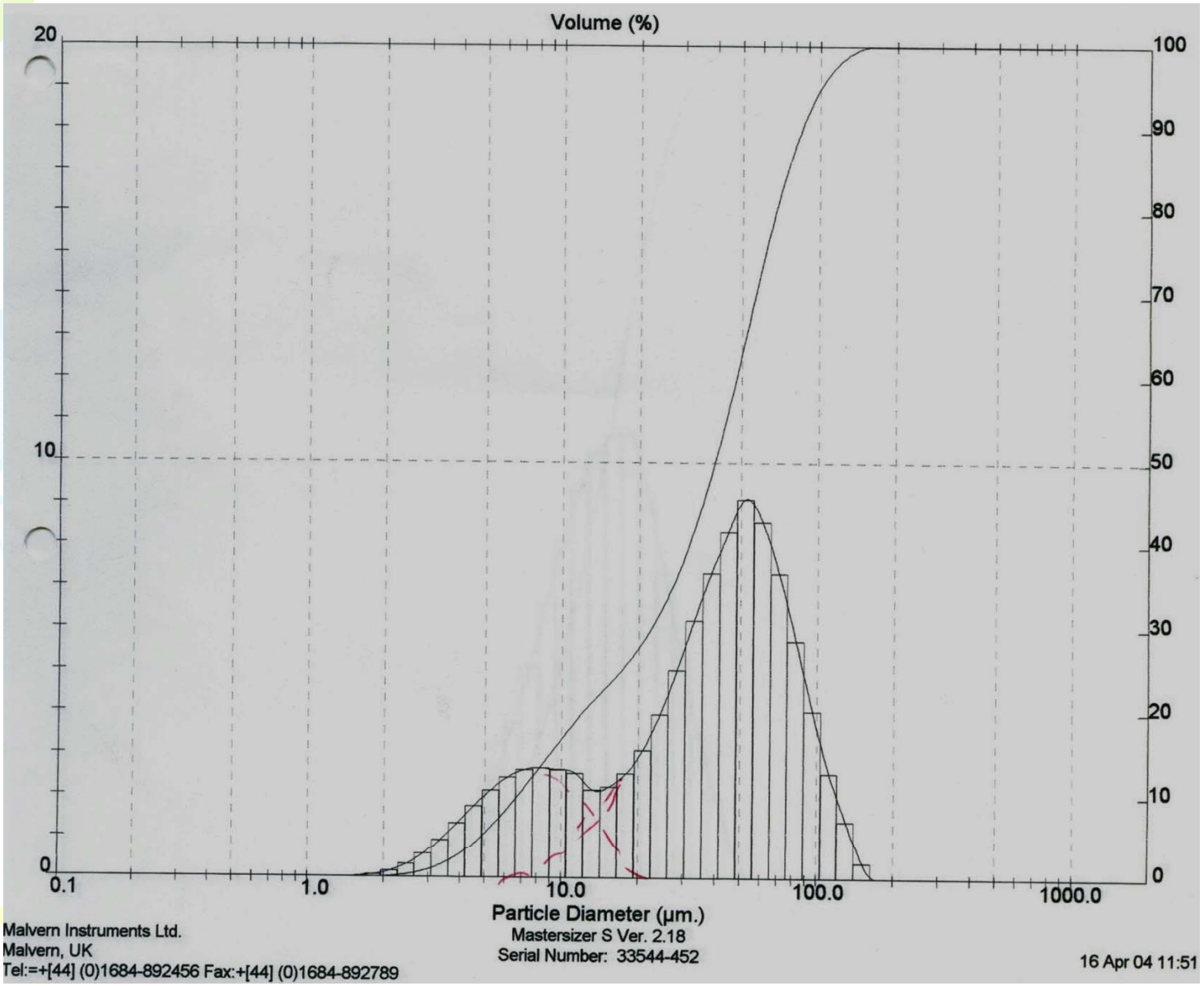
Parameters Affecting the Properties

- Concentration of silicate in the solution
- Rate of carbonation
- Agitation of the solution while carbonation
  - Design of the stirrer and its location relative to the CO_2 injection ports
- Mode of CO_2 injection—velocity of injection, number and distribution of injection holes
- Extent of carbonation

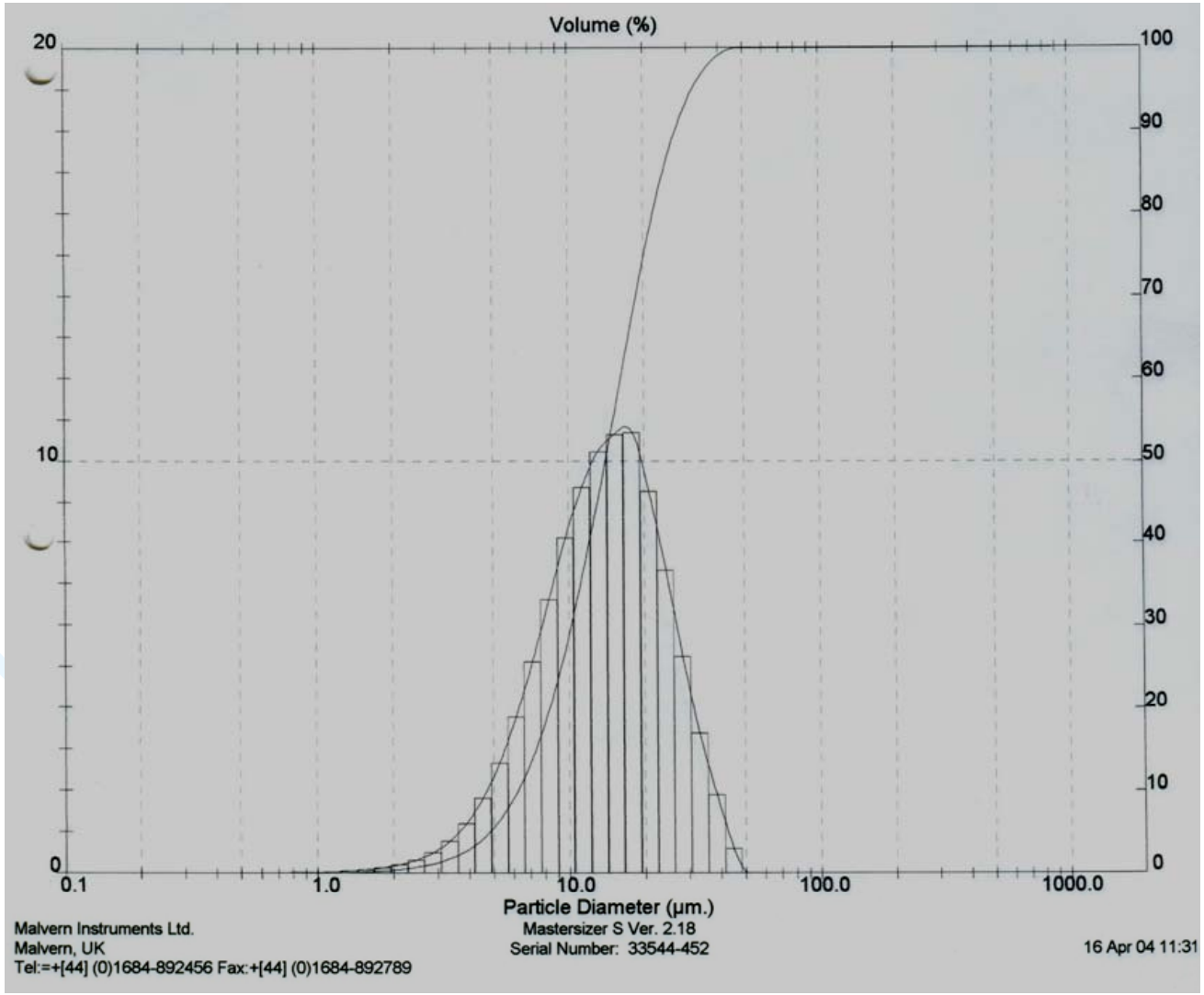
Comparison of IPSIT silica with VN3

Serial No	Particulars	Unit	VN3 Range as per NIPSIL	VN3 Lot No 3BU11 - Specs	VN3 as per Lot No 3BU11- Analysis - IISc	I.I.sc. Silica
1	Weight	Kg		20	Received - 5 kg	
2	Water Content	%	9.0 or below	4.6	5.20%	4.50%
3	PH	4% Susp.	5.5 - 6.5	6.0	5.23	6.6
4	Tap Density	g/l	120 - 160	152.0	260	175
5	BET Specific Surface Area	m2/g	180 - 230	220.0	217	229
6	Particle Size(150µabove) by Sieve	%	7 or below	1.2	**90 % - 84 microns**	**90 % - 27 microns**
					50 % - 39 microns	**50 % - 14 microns**
					10 % - 6.74 microns	**10 % - 6.2 microns**

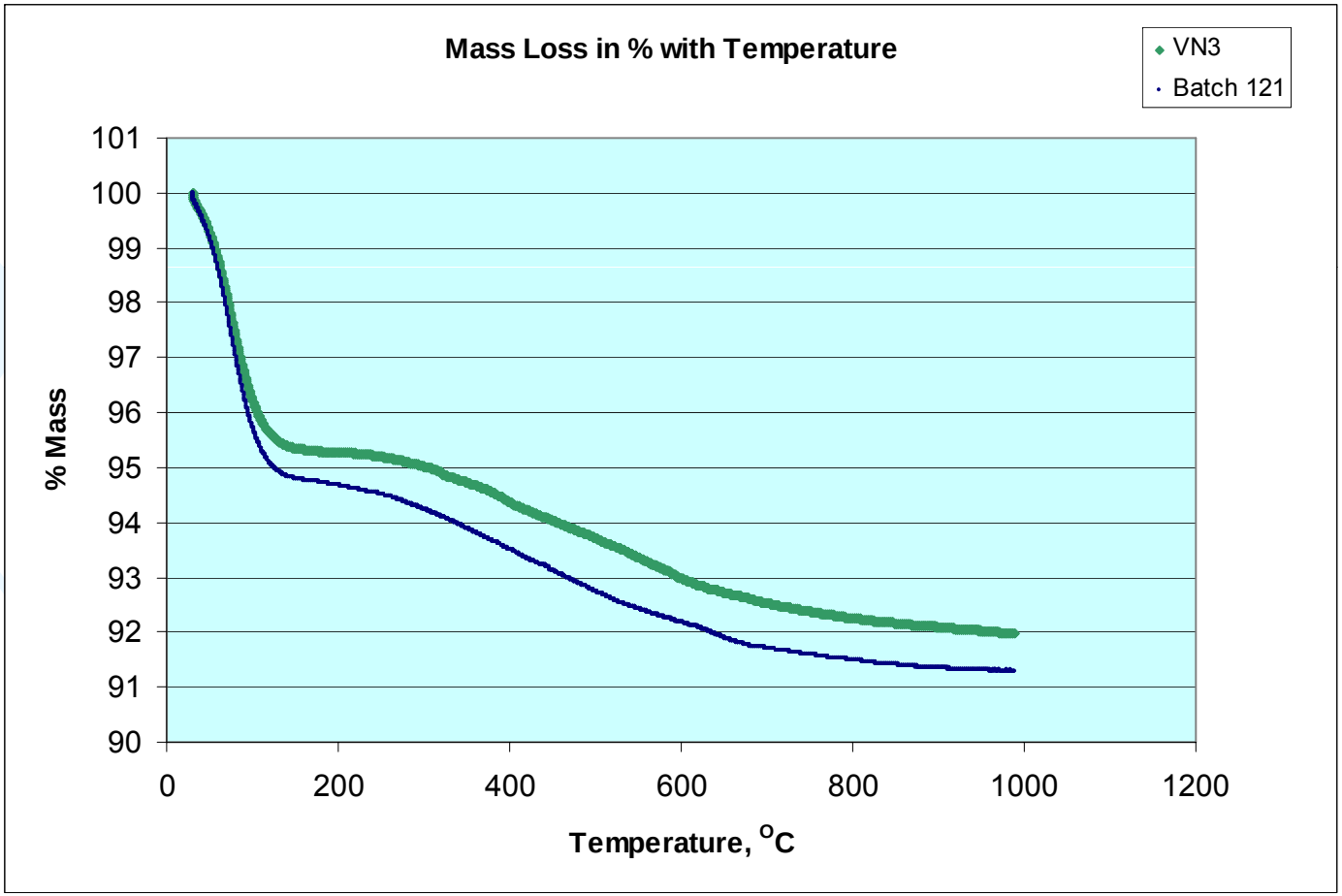
VN3 – Particle Size Distribution



IISc Silica Particle Size Distribution



TGA Analysis of VN3 and IISc Silica



Concluding Remarks

- Ready for commercial launch.
- Licensed to IVY – Japanese form
- Pilot plant of 50 kg/day is being set up along with IVY – Biosilica
- Commercial plant of around 24 TPD to be set up later

Thank you