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Pilot Plant Experiments for Extraction of Inorganic Products from Agro Solid Waste and Cost Estimation of the Products

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Abstract

The present article aimed to synthesize spherical silica nanoparticles (SNPs)from rice husk ash as an inorganic product, and to estimate cost of the products production. SNPs were prepared in pilot scale from RHA using optimum operating conditions, resulted from laboratory experiments (2.0 N NaOH, 100 $^{\circ}$ C, 2h reaction time and stirring rate 120 rpm). The produced SNPs are characterized by sharp peaks of silica, amorphous spherical shapes, particle sizes of 18-75 nm and surface area 200 m²/gm. The manufacture of the target product SNPs is thoroughly investigated economically, taking into consideration the production of sodium sulfate fertilizer as by product and using RHA in bricks manufacturing. Techno-economic study was carried out for productivity of 5400 ton/year of silica, 60,264 ton/year fertilizer, 7,800,000 sand brick unit/year.

Keywords: Rice husk ash, nano silica preparation, process design, cost estimation, fertilizers

1. Introduction

Rice Husk (RH) is one of the most widely available agricultural solid waste residues in Egypt. It is a byproduct of the industrial processing of rice and 20wt.% of bulk grain weight. RH is utilized as fertilizers, insulation materials, fuel, filler, catalyst support, adsorbent and the rest is burned in an ambient atmosphere to form RH ash (RHA), which represents an economic and environmental burden on Egypt as it mainly causes toxic air pollution. The uncontrolled combustion of RH results in enormous ecological threats, causing damage to the land and surrounding area in which it is dumped. Hence, maximizing the reuse of this waste will be vital to any solid-waste management strategy [1-3]. RH yield on burning RHA with 85-95% silica content, and the rest is some carbon and other nonmetallic and metallic impurities. Controlled burning influences the purity and color of the ash as reported by many authors as it decreases metallic ions and produces silica in white amorphous or crystalline silica form of high purity [4-6]. Bench scale experiments was carried out by the present authors [7-9], where alkalization method was used to produce SNPs from RHA to be used as adsorbent materials for dyes and pigments pollutants in industrial wastewater. The objective of the present article is to: 1) prepare silica nanoparticles (SNPs) from calcined rice husk (CRH) on pilot scale based on optimum operating conditions

determined from the bench scale experiments and 2) carry out a techno-economic study for the innovating SNPs adsorbent resulted from pilot experiments to support and guide decision-makers in their assessment for this locally manufactured product.

2. Materials and Equipment.

2.1. Materials

Rice Husk (RH) from El-Shariq Governorate in Egypt is selected for the present investigation. Analytical grade of sulfuric acid (95%-98%) and sodium hydroxide (NaOH) (Aldrich) were purchased from El-Nasr Company for Chemicals, Egypt.

2.2. Equipment

2.2.1. Muffle furnace:

Programmable muffle furnace controlled by high precision temperature controller with +/- 1^oC accuracy and 40 programmable segments and furnace housing built with double-layer steel is used for calcination process.

2.2.2. Pilot plant set-up:

A pilot reactor set-up, equipped with auxiliaries, was fabricated locally to carry out the pilot experiments for preparation of SNPs (Fig.1). It consists of a double jacketed reactor of 12 L capacity, from St-St. 304 for heating process. The inner dimensions of the reactor are 24 cm length x 30 cm diameter, supported

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on four metallic legs of 80 cm height to allow suitable reactants discharge. There are two openings of 1 in diameter for charging and sampling of reactants. The reactor top is equipped with a condenser and a mechanical variable stirrer up to 500 rpm. Further, the reactor is equipped with a control system for measuring and controlling the temperatures of the heating fluid and the reactants mixtur



Fig.1. Pilot plant setup

2.2.3. Methodology 2.2.3.1. Experimental Procedure Preparation of CRH

The foundation of the merits for this proposed process among others alternative processes is first the rice husk ash with higher silica content. Initially, the process to extract all the silicate contents from the rice husk - being an agricultural waste and procured directly from the mill in its original form - is just by burning it under controlled conditions of low air flowrate level and temperature, in a programmable muffle furnace directly at 500 $^{\rm o}$ C for 2 hours, at a heating rate of 10°C/min which are optimum conditions resulted from a previous parametric study carried out by the present authors on laboratory scale via the 1st progress report of the Project NO 30455 Funded by STDF, Egypt [8]. The laboratory studied parameters were 450-700 °C calcination temperature, 1-3 hour calcination time, and heating rate of 5-15 ^oC/min. By this condition, we can get the clear white ash in the furnace.

Preparation of SNPs on pilot scale

This ash is mostly amorphous silica which is reactive with NaOH solution at low temperature and atmospheric pressure to yield sodium silicate. Two kilograms of prepared CRH (rice husk ash) is dispersed in a stoichiometric amount of 2N NaOH aqueous solution and heated at 100 °C for 2 hours under vigorous stirring at 100 rpm stirring rate to dissolve silica and produce sodium silicate, according to Eq.1. These conditions was the optimum conditions resulted from previous laboratory experiments carried out by the authors of the present article [9,10], (the parameters studied in laboratory experiments were stirring rate 120-500 rpm, 1-3N NaOH, heating temperature 20-100 time of extraction 1-3 hour. Removal of free hydroxide were carried out by neutralization of the unreacted NaOH by H₂SO₄ solution For reproducibility check, pilot plant experiments were conducted twice.

A viscous, transparent, colorless sodium silicate solution is obtained after filtration of the reacted slurry and washing to remove the nonreactive impurities, comprising mainly residual digested ash. The transparent filtrate of sodium silicate solution was allowed to cool to room temperature before

Na₂SiO₃ + (sodium silicate) H_2SO_4 (sulfuric acid)

Thus, sodium silicate solution is slowly neutralized under controlled conditions of addition rate with 8N sulfuric acid solution to precipitate silica (SiO₂.H₂O). A gel was formed during the reaction at pH values of 7,8 and 9, and the obtained suspension was next filtrated, followed by washing the yellowish gel repetitively with water until no sodium sulfate was detected in the wash water. Then, the spent solution is directed to sodium sulfate recovery unit as added value to the process while the clean silica gel cake is next dried in an oven at 100 °C to minimize its water content till constant weight. The final dried silica appeared as fine and white powder with purity 87.2%.

2.2.3.2. Characterization and examination tests

Physical and chemical analyses of the silica produced (SNPs) was investigated. The structure of the produced SNPs was studied by X-ray diffraction (XRD), while X-ray fluorescence (XRF) is used for

further processing. In the final step of the process, precipitation of the silica nanoparticles from sodium silicate solution was induced through the addition of sulfuric acid under stirring at normal atmospheric pressure as follows:

$$SiO_2$$
 + Na_2SO_4 + H_2O (2)
(silica) (sodium sulfate) (water)

chemical analysis. The morphology of the produced SNPs was examined by Electron Transmission Microscopy (TEM) and Scan Electron Microscopy (SEM). Fourier Transform Infrared Spectrometry (FTIR) analyses is used to identify the chemical bonds. The specific surface area of the SNPs was evaluated using the BET method, and the pore size (the pore volume and the pore radius) distribution was calculated by the BJH and TPV methods. The residue obtained from the filtration step of sodium silicate also analyze.

3. Results of the prepared SNPS

3.1. Product appearance

The appearance of: (a) Sodium sulfate formed, (b) residue of CRH, (c) silica gel and (d) silica nanoparticle (SNPs) yield all over the process of silica production is presented in (Fig.2)



(c) Filtered Silica gel

(d) SNPs

(a) Sodium sulfate



(b)CRH residue

Fig.2. Products appearance (a, b, c, d)

3.2. X-ray fluorescence (XRF)

Table 1 depicts the XRF of raw RH, the residue of the filtration step and the silica yield resulted from the pilot plant experiments. It can be concluded that the extracted silica was white amorphous powder, composed of 87.82% SiO₂, 2.36% metal oxides impurities and 10.17% loss of ignition.

3.3. XRD of SNPs

The XRD (Fig.3) expresses the presence of amorphous silica only and there is no presence of quartz. Fluctuating small peaks of amorphous silica are observed from the XRD pattern of SNPs (Fig.3). In 4 theta positions: 1) $2\theta = 21^{\circ}$ of intensity count

9.5, 2) $2\theta = 23^{\circ}$ of intensity count 10.5, 3) $2\theta = 23.5^{\circ}$ of intensity count 9.7 and 4) $2\theta = 26.339^{\circ}$ at intensity counts of 7.51. The main peaks are in the range of $[2\theta] = 21^{\circ}$ to 26.339°, confirming that the SNPs formed are mainly amorphous silica also indicated the absence of any ordered crystalline structure

3.4. Scan Electron Microscopic Examination (SEM) of SNPs)

Fig.4 (a), (b) presents the surface morphology (SEM) of the SNPs at magnification of 40,000X and 80,0000X respectively. It is observed that SNPs obtained are in amorphous form with spherical shapes. Due to the nonconductive nature of silica, the

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charges	quickly accumu	late	d on the p	owd	er surfaces	
caused	agglomeration	of	particles	as	previously	

reported [10].

	Table	e 1: XRF	results of	raw RH,	residue	of filtrati	on step	and SNF	S	
Metal Oxides%	SiO ₂	Al ₂ O ₃	Fe_2O_3	MgO	CaO	Na ₂ O	K ₂ O	P ₂ O ₅	Other traces of metal oxides	LOI
Raw RH	18.09	0.11	0.06	0.10	0.16	0.10	0.20	0.07	traces	80.87
Filtration Residue	45.82	4.21	12.17	2.99	11.3	0.48	1.11	1.06	19.00	18.09
SNPs	87.82	0.35	0.08	0.11	0.14	1.09	0.12	0.01	0.11	10.17
21 20 19 18 17 16 16										



Fig.3 XRD of Silica Nano Particles



(a) SEM of SNPs at magnificent of 40,000X

(b) SEM of SNPs at magnificent of 80,000X

Fig.4 (a), (b) the surface morphology (SEM) of the SNPs

3.5. Transmission Electron Microscopic Examination (TEM) of SNPs

TEM examination of the SNPs (Fig.5 (a), (b)), proved spherical particle sizes of 18-75 nm.



(a)TEM SNPs

(b) TEM SNPs



3.6. FTIR of SNPs

The FTIR spectrum of optimum SNPs produced (Fig.6) showed that the vibration signals around 1063.60, 962.95, 796.77, 557.77, 551.67.35 and 451.67 cm-1 which are typical of Si-O-Si bands attributed to the symmetric stretching and bending, respectively [11]. These six peaks are the main indices of the silica materials, which represent the successful production of SNPs. The absorption band for H-O-H bending vibration in water was at 3400.6 and 1632.80 cm⁻¹, which resulted from the presence of the O-H stretching frequency for the silanol group and the remaining adsorbed water. No peaks were found between 3400.6 and 1632.80 cm⁻¹. It means that there were no original organic compounds in the silica after controlled combustion and extraction which in good agreement with the published data [12,13].

Table 2: BET and BJH of optimum extracted SNPs.



Fig. 6 FTIR Spectra of optimum extracted SNPs

3.7 BET and BJH of SNPs

BET and BJH of the silica yield are presented in Table 2. The surface area of the spherical particles of SNPs is $200 \text{ m}^2/\text{g}$ of average pore radius of 3.99 nm.

BET	BJH (pore sized distribution)				TPV (total pore volume)
(APR) Average pore radius	Surface area	Surface area	Pore volume	Pore radius	-
3.99 nm	200.075 m ² /g	125.192 m ² /g	1.06 cc/g	1.93 nm	Total Pore volume 1.1012e ⁺⁰⁰⁰ cc/g for pores smaller than 100.38 mm (radius) at relative pressure 0.99033

The properties and the constituents of the RH and RHA are found to vary from place to place. The type of paddy, climate, and geographical conditions, in addition to the sample preparation and method of analysis, could be the reason for this variation. The XRF of the Egyptian raw RH, indicates that the SiO₂ content is 18.09%, the summation of metal oxides (Al₂O₃, Fe₂O₃, MgO. Cabo, Na₂O, P₂O₅,......) are 1.04% and the loss of ignition (LOI) is 80.87%. After

calcination, the silica and metal oxides contents increase to about 92% and 6% respectively while the LOI decreases to 2%. It is believed that these impurities interfere in the alkalization reaction and which affects the purity of the final product. For that further purification will be costly

4. Techno-economic study: process design and cost estimation

Details of the process design to produce of 5400 ton/year of SNPs from rice husk waste disposal are presented, covered the process description, material balance, energy requirements specifications and selection of principal equipment. In addition, a preliminary economic study is reported.

4.1. Process Design

4.2. Process Description

The proposed process is a new process where silica is manufactured by means of the alkalization of rice husk ash as a source of silicate or silica with sodium hydroxide which yields the sodium silicate, which on further neutralization with sulfuric acid produces silica along with a by-product sodium sulphate. Hence, three basic steps as described in item 3.2. a) Obtaining silica from rice husk, b) Dissolution of silica in alkali and c) Preparation of silica from silicate solution. A qualitative block-type process flow sheet is illustrated in Fig.7

4.2.1. Material balance

Several batches of calcined rice husk are carried out apart and considered as a separate entity from the process. According to data obtained from pilot experiments, material balance calculations are first carried out based on 1kg RHA, then, was scaled up based on the following data:

- The productivity: 5400 ton/year (10% moisture content).
- Plant operation: 300 days/year.
- Daily design production rate: 18 ton/ day (10% moisture content).
- Daily design working shifts: 2 shifts/day (8 hours/ shift).
- Daily operating batches for RH calcination: 5 batches/day
- Daily operating batches for RHA processing: 9 batches/ day
- Design production rate: 2 ton /batch
- Product solid content: 1.8 ton solid/ batch
- \blacktriangleright RH required for calcination =144ton/day
- \blacktriangleright RHA produced =3.422 ton / batch.

The mass balance flow sheet to produce 2-ton silica/batch is shown in (Fig.8). While, energy balances (Fig.9) illustrated of design for some specific equipment as reported elsewhere [14].

4.2.2. Equipment Sizing and Selection

The process is essentially batch even though the drying step and sulphate concentration are continuous. For economic considerations, the process is planned and developed as to operate according to a proposed time schedule- relevant to individual stepwise residence time-, namely, nine batches per day with one hour interval each (Fig.10). Two reactors for alkalization are designed to feed the first filtration step alternatively with one hour interval each. Equipment flow sheet (Fig. 11) was prepared to obtain a systematic organization of sizing calculations. This is coded by letter for each key operation and all equipment conveniently associated with the key operation is numbered.

5. Preliminary cost estimation

Economic indicators of the target product in nanoparticles form, namely silica extracted from rice husk is presented in this section. Production costs are basically divided into two categories; total fixed capital costs- incurred during plant construction- and annual operating costs necessary to provide sustained operation for the plant after construction. Data of fixed capital costs and annual operating costs were obtained from reliable sources. For the sake of profitability analysis, the product cost of both silica and the by –products sulfate fertilizer and rice husk bricks are estimated separately.

5.1. Fixed-capital and Annual operating costs of silica extraction from rice husk.

Fixed-capital cost estimation requires determination of the purchased- equipment cost. The other included items are then estimated as percentages of the purchased –equipment costs. Costs of equipment locally purchased are estimated according to current costs as provided by local market, while imported equipment are estimated according to international firm suppliers included freight and customs charges (1 = 15.7 L.E. based on January year 2021). Values of the various percentages used in estimating the fixed-capital investment (F.C.I) [15] along with proportional costs of major components of total capital investment (T.C.I.) are demonstrated in Table 3.

Furthermore, the various cost elements, directly connected with the manufacturing operation are presented in Table 4. The obtained prices of raw materials and utilities are: *-Raw materials* (commercial grade): Sodium hydroxide (17LE/Kg), conc. sulfuric acid (5 LE/kg). It should be noticed herein that the raw rice husk is considered as a priceless agricultural waste with nil cost. *-Utilities*: electricity (4L.E./kW), steam (105 L.E./ton), process water (6 L.E./m³) and cooling water (2.6 L.E./m³).



Fig.7 Block flow diagram of silica (SNPs)production from rice husk ash

Table 3 Estimation of Total Capital Investment for silica production from rice husk

Components	Price, in 1000 LE
I-Direct Cost:	
1-Purchased equipment cost (E)	11201
2-Purchased equipment installation (30%E)	3360
3-Instrumentation and control (13%E)	1456
4-Piping (30%E)	3360
5-Electrical equipment and materials (8%E)	896
6-Buildings (including services) (18%E)	2016
7-Services facilities & Yard improvement (30%E)	3360
6-Land (6%E)	672
Total direct cost (D)	26,321

II-Indirect cost

1-Engineering & Supervision (5%D)

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2-Constructions expenses & Contractor's fee ((7%D)	1842
3-Contingency (10% F.C.I.)		3276
Total indirect cost (ID)	6434
III-Fixed-capital investment (F.C.I.)		32,755
IV-Working capital (15% T.C.I.)		5780
V-Total capital investment (T.C.I.)		38,535

Table 4 Annual operating cost for silica production from rice husk

Component	Annual price in 1000 LE
1-Raw materials	
Sodium hydroxide	193,375
Sulfuric acid	69,710
Total raw materials	263,085
2-Utilities	
Electricity	2862
Process water	980
• Steam	3850
 Cooling water (35 % make-up) 	192
Total utilities	7,884
3-Maintenance (3% for buildings and 5% for installed equipment)	620
4- Operating labor (29,700 man-hours /year) *	371
5- Laboratory charges (10% of operating labor)	37
6-Administrative expenses (20% of operating labor)	74
Total Annual Operating Cost	271,451

*Operating labor is estimated based on three main production steps and 5000 LE/month. labor as average salary including incentives.

5.2. Fixed-capital and Annual operating costs of sodium sulfate solution (by-product liquid fertilizer). As mentioned above, the equipment cost relevant to the concentration of spent sodium sulfate solution from 12.94% $_{wt/v}$ to reach 40% $_{wt/v}$ to be used as soil

fertilizer and the reused of recovered water in the process, is excluded from the above cost evaluation. Therefore, Tables 5,6 depict the capital investment and the annual operating cost to estimate the fertilizer solution product cost.

Table 5 Estimation of Total Capital Investment for fertilizer production

Components	Price, in 1000 LE
I-Direct Cost:	
1-Purchased equipment cost (E)	2,291.24
2-Purchased equipment installation (20%E)	458.25
3-Instrumentation and control (5%E)	114.56
4-Piping (15%E)	343.68
5-Electrical equipment and materials (3%E)	68.74
6-Buildings (including services)	
7-Services facilities & Yard improvement (20%E)	458.25
6-Land (6%E)	137.47
Total direct cost (D)	3,872.20

PILOT PLANT EXPERIMENTS FOR EXTRACTION OF INORGANIC PRODUCTS......

1-Engineering & Supervision (3%D)	116.17
2-Constructions expenses & Contractor's fee (3%D)	116.17
3-Contingency (10% F.C.I.)	456.06
Total indirect cost (ID)	688.40
III-Fixed-capital investment (F.C.I.)	4,560.60
IV-Working capital (15% T.C.I.)	804.80
V-Total capital investment (T.C.I.)	5,365.40

Table 6 Annual operating costs for fertilizer production

Component	Annual price in 1000 LE
1-Total raw materials	
2-Utilities	
Electricity	205
• Steam	14,727
Cooling water	11,804
Total utilities	26,736
3-Maintenance (1% for concrete equipment and 2% for installed	54.95
equipment)	
4- Operating labor (10,000 man-hours /year)	125
5- Laboratory charges (10% of operating labor)	12.5
6-Administrative expenses (20% of operating labor)	25
Total Annual Operating Cost	26,954

5.3. Cost estimation of bricks by-product production

A previous comparative estimation of the unit cost of the modified RHA–sand/lime and cement/RHA bricks, which had been earlier prepared and reported by the authors, was performed [16]. It was concluded that the partial replacement of RH, as solid agricultural waste, in the manufacture of building bricks can reduce the cost significantly, as 20% RH substitution (yielding 5% RHA) in the sand brick, and also in the cement brick, resulting in 14% and 25% -unit cost reductions respectively.

According to data obtained from the previous study, and taking into consideration the Marshall and Swift indexes, the cost estimates parameters of the sand/rice husk ash bricks and the cement / rice husk ash bricks produced from the present residual RHA [5384 ton / year (45.8% SiO₂)] are evaluated.

5.4. Unit Production cost

The production cost was performed based on the final composition resulted from the calcination of RH. The course of the material flow in the production process can be followed in the material balance flow diagram presented (Fig.8).

The production cost is the sum of total annual operating cost and total depreciation rate per unit product capacity. Annual depreciation rate is estimated based on a useful-life period of 10 years of the fixed capital cost excluding buildings and concrete equipment, while buildings and concrete equipment are depreciated based on 30 years lifetime. Tables 7, 8 depict the cost estimates parameters of the SNPs and by-products, extracted from rice husk.

Table / Estimated cost parameters of extracted since and sodium suppliate fertilizer (40% conc.)					
Product item	Annual	Annual operating	Annual	Unit product cost,	
	production,	cost, LE/year depreciation		LE/Kg	
	ton/year		LE/year		
SNPs	5400	271,451,000	3,342,700	50	
Fertilizer	60,264	26,954,000	395,260	0.5	
Table 8 Cost estimation of	of building bricks	* by-products			
Product item	Anı	nual RHA residue,	Annual production	. Product cost,	
		ton/year	bricks/year	LE/10 ³ bricks	
Sand/rice husk ash	า	4470	7,800,000	730	
Cement / rice husk a	ish	914	7,600,000	855	

Table 7 Estimated cost parameters of extracted silica and sodium sulphate fertilizer (40% conc.)

* Brick dimensions: 25cm x12cm x 6cm.

5.5. Profitability analysis

In a broad sense, the profitability is the determination of the relationship between income and expenses so that a sound decision can be taken to whether a given project should be financed or not. Two of the most used methods for profitability evaluation are the determination of the rate of return on investment (*ROI*) and the payout period with no interest charge.

As a performance measure, *ROI* is used to evaluate the efficiency of an investment, via the following expression:

% Return on Investment (ROI) = [Profit / (T.C.I.)] x 100 A payout can refer to the period in which an investment or a project is expected to recoup its initial fixed capital investment, via the following expression:

Payout period= (*F.C.I.*) / (*Net profit* + *Depreciation rate*)

Table 9 demonstrates the cost and profit summary for silica production from rice husk as principal product, in addition to its fertilizer by-product solution and building bricks products as added values. Sales price are estimated in view of market competition as follows:-*Silica* = 65 LE/Kg; *Sodium sulfate fertilizer* (40%) = 1.5 LE/Kg; *Sand/RHA bricks* = 800 LE/10³ bricks and *Cement / rice RHA bricks* = 900 LE/10³ bricks

Total products cost, LE/year	340,892,000
Total products value, LE/year	454,480,000
Gross profit, LE/year	113,588,000
Net profit*, LE/year	54,522,240
T.C.I., LE	59,288,600
% ROI	92
Pay-out time, year	1.5

6. Conclusion

Green production philosophies suggest designing a process having a commercial value for its waste products. From this point, the production of silica particles in nano form, (SNPs) from rice husk (RH), being an agricultural waste from rice cultivation in Egypt - over one million acres of rice crops were grown in Egypt, producing 1.6 million tons per acre of husks after the harvest in October and Novemberis carried-out on pilot plant scale, by the alkalization method, aiming to be used as adsorbent material for treatment. Characterization wastewater and assessment of the silica product and its indigestive residue are conducted by physical and chemical analysis. The manufacture of the target product is thoroughly investigated economically, taking into consideration the production of three by-products, yielding from the process as added values. The resultant economic indicators assure the inevitability of maximizing the utilization of agricultural rice residues in using them to produce materials of economic value and make the current study useful for governmental decision makers, by assuring an efficient revenue generation achievement while ensuring utilization of the waste generated.

Declaration of Competition Interest

The authors declare no conflicts of interest.

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Fig. 8 Material balance of SNP production from rice husk ash. (Basic: 2ton silica/ batch)



Fig.9 Energy flow diagram of silica gel production from rice husk ash. (Basic: 2ton silica/ batch)

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Fig.10 Sequential operation scheme for SNPs production



Fig.11 Equipment flow sheet for silica

production