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Photovoltaic solar cells industry wastewater treatment

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ABSTRACT

Nowadays, in the photovoltaic (PV) industry there still remains a huge potential to be exploited, where markets are dominated by crystalline silicon PV-based cells. However, in the future it is expected that thin films PV will have a larger market share. Until recently, the prevailing technology based on mono-crystalline silicon has been gradually exceeded by poly-silicon technology due to its lower production cost. In PV industry, solar-graded silicon is the main material used in manufacturing process. In this industry, manufacturing and production processes require large amounts of water that result in important discharged industrial effluents containing different pollutants such as hydrogen fluorides, suspended solids, mixed acids, SiO_2, and high oxide particles. Among discharged pollutants, the hydrofluoric acid is significantly used in photovoltaic's (PV) manufacturing for both quartz cleaning and wafer etching. In fact, wastewaters from PV industries have high concentrations of fluoride, typically in a range of 500–2,000 mg/L. They are considered highly toxic and need to be strictly monitored and regulated. Three production phases that generate the highest flow of wastewaters are texturing (multi-crystalline silicon wafers need a mixture of HF/HNO_3 dilution and also a hot caustic solution with IPA used for the multi-crystalline wafers mixture), etching, and formation of PV cells. Phosphorus diffusion leads to formation of phosphorus silicate layer that needs to be eliminated by means of hydrofluoric acid (HF) application. Classification of effluents from a point of source, concentration, chemical, or composition feature is compared. Wastewater treatment optimization is often conducted and we discussed major treatment methods in solar cells manufacturing: treatment of HF discharges, neutralization, and collection of isopropanol discharges. The article discusses design of wastewater treatment system that is operational in practice.

Keywords: Solar cell; Silicon wafers; Treatment of hydrofluoric acid; Isopropanol discharges; Neutralization; Sedimentation; Filtration

1. Introduction

The sun represents an almost inexhaustible source of energy. The challenge has always been to capture this energy and transform it into electricity.

The solar photovoltaic branch industry is growing rapidly and electricity production from solar energy is becoming more efficient and less costly thanks to innovations in technology. Although, the proportion

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of electricity produced from solar cells is continuously increasing, environmental concerns are unavoidable in the choice of industrial products [1].

Solar cells are manufactured in a complex process that requires enormous know-how. The objective is to produce panels with high level of efficiency at low cost. To achieve this, different production processes are used with a fundamental distinction between solar cells on the basis of silicon wafers and thin-film cells. The latter technology, known as the second-generation PV cells relies on special process to apply the photovoltaic layer onto a carrier medium. Photovoltaic cells manufacturers of solar cells are constantly developing and improving production processes. Regardless of technology used, large quantities of water are required and this eventually results with polluted wastewaters. Water is becoming increasingly valuable as a raw material necessitating efficient water management of this resource. The wastewater from the production process must be treated in a way to recycle as much water as possible. The treated wastewater must comply with discharge parameters in order to prevent pollutants going into environment. For this reason, optimization of the production process and the wastewater treatment are often necessary [2].

Environmental and economic impacts associated with the photovoltaic systems are mainly due to hydrofluoric acid (HF) effluents generated during the various steps of fabrication.

This article provides an overview for the typical wastewater techniques for silicon solar cell manufacturing. The main steps of the process are described as well as types of waste generated during production. Methods of the treatment of HF streams are presented. In addition, few options for the water reclamation and reuse are discussed and guidelines for the design of wastewater treatment systems are given.

2. Production process

2.1. Description

Solar cells are produced from solar grade silicon (SOG). Silicon is manufactured by a silica reduction process in an arc furnace. Metallurgical quality silicon is obtained in this way, containing around 1% impurities. The silicon then solidifies ejecting metallic impurities. After that, it is purified using a plasma torch to remove any boron.

The crystallization of the silicon is performed by extracting heat through the base of the container that allows to the crystals to grow in a controlled way. The most performed solar panels are manufactured from the larger crystals that also may contain a fewer defects [3].

Using various saws, ingots produced are cut into bricks and then into wafers. The wafers are attacked by an acid or base to reduce their reflectivity.

In the diffusion furnace, phosphorous atoms are added to the surface of the cells creating an electric field where electrons will be collected. A nitride deposit then allows to the reflectivity to be improved further. Contacts are deposed by screen printing that allows to the electric charges to be collected. Cells are then annealed to eliminate solvents and ensure the mechanical strength.

Finally, a laser is used to isolate the junction. The finished cells are then assembled into modules that are characterized and tested under real sunlight.

2.2. Types of effluents generated: characteristic and classification

Photovoltaic cells production generates different types of effluents from successive processes steps:

- Crystalline growth (elaboration of multi-crystalline silicon wafers)
- Silicon wafers sawing
- Texturing
- Phosphorous doping
- Etching
- Metallization

Some processes steps generate low volumes of effluents, while others produce significant volumes of rinsing water and concentrated acids that must be treated. The process steps that generate the most significant flux and concentration of wastewater are the following (Fig. 1):

- Texturing
- Phosphorous doping
- Photovoltaic cell formation

Typical characteristics of wastewater resulting from photovoltaic solar cells wastewater plant are summarized in Table 1.

2.2.1. Texturing

Depending on the type of silicon wafers, several methods are used for the sawing defects elimination (texturing process):

- The multi-crystalline silicon wafers need a mixture of hydrofluoric and nitric acids diluted (HF/HNO_3).

Table 1
Operational flow for silicon solar cell plant

Type of wastewater	Flow
HF-C	217.4 l/h
HF (49%)	63.3 l/h
HCl (37%)	4.2 l/h
HNO_3 (65%)	78.3 l/h
HF-D	6.275 l/h
CAW	2.531 l/h
IWW	508 l/h
PreWaCleaner Si 021	1.5 l/h
CW-D	2.084 l/h
Si	2.90 g/l
SiC	0.26 g/l
PEG	3.67 g/l
CH_3COOH	1.79 g/l
InWaCleaner Si 011	20.0 l/h
CT-BW	480 l/h
SiO_2	2.9 g/l

HF: hydrofluoric acid; HNO_3: nitric acid; HCl: hydrochloric acid; NaOH: sodium hydroxide; NaOCl: sodium hypochlorite; Si: silicon; SiC: silicon carbide; PEG: polyethylene glycol; HF-D: HF diluted; HF-C: HF concentrated; AWC: acid wastewater concentrated; CAW: caustic wastewater; CW-D: caustic wastewater diluted; IWW: industrial wastewater.

This so called buffer edge solution is needed to eliminate defects in the crystal structure caused by the manufacturing process of the wafers (on the surface of each wafer). During the process, chemical baths are doped in order to maintain the quality of the etching solution constant [1–5]. Nevertheless, the bath should be completely changed at regular intervals, depending on consumption. This requirement leads to a steady flow of HF/HNO_3 solutions at a high concentration, which in turn require further treatment.

- For mono-crystalline silicon wafers, the process is usually performed using a mixture of hot caustic solution with IPA.
- As for poly-crystalline silicon wafers, the baths used in this process must be completely changed at regular intervals and for the same reasons.

Concentrated flows of isopropanol (IPA) are problematic, primarily due to their high temperature, so they need to be cooled before they are collected for the final discharge.

2.2.2. Phosphorous doping

During the doping process, phosphorus is used to diffuse into the substrate doped with boron in order to create the p/n junction in silicon wafer. The phosphorus diffusion process leads to the formation of a layer of phosphorus silicate over the surface of the silicon wafer, which must be eliminated through the application of a dilute solution of hydrofluoric acid (HF). This HF must be treated in the wastewater treatment installation [1].

2.2.3. Photovoltaic cell formation

The process of deposition of silicon nitride is carried out with the aim of reducing the light reflection on the wafers surface. The treatment chamber must be cleaned regularly (for example with an exhaust system for fluoride). Treatment of this evacuation is usually performed by reduction of local systems. It can generate a small amount of wastewater, but the level is relatively low compared to the first three processes. The other production process steps usually generate little or no wastewater [6].

2.2.4. Effluent characteristics

Wastewater from photovoltaic wafers production industry contains HF, mixed acid etchant, suspended solid (SS), turbidity, SiO_2, and high oxide particle. The pH of wastewater is between 2 and 4.

2.2.5. Effluent classification

Different types of wastewaters are generated during the production of photovoltaic solar cells which are made from crystalline silicon. Distinction can be made according to their source (bathing, cleaning), concentration (diluted, concentrated), the chemical (acids, bases), or according to their compositions (containing fluoride, not containing fluoride) [7,8].

In practice, different types of wastewaters are classified according to chemical concentration and composition. Wastewaters from the production of photovoltaic cells are generally classified into two groups of wastewater:

- Rinsing effluent: the rinsing water, which has much lower concentrations of chemicals, is treated *in situ* in wastewater treatment system.
- Concentrated acids effluents: strong acids are generally collected for external discharge.

From the perspective of the composition, the level of fluoride content is one of the most important parameters to be considered because of its relatively strict discharge concentration limit.

Fig. 1 shows the process steps and types of wastewater streams generated during the production of

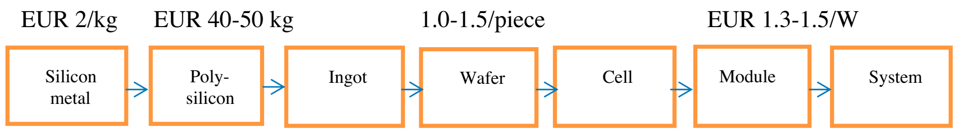

Fig. 1. Breaking down production process of solar modules with pricing information for various components.

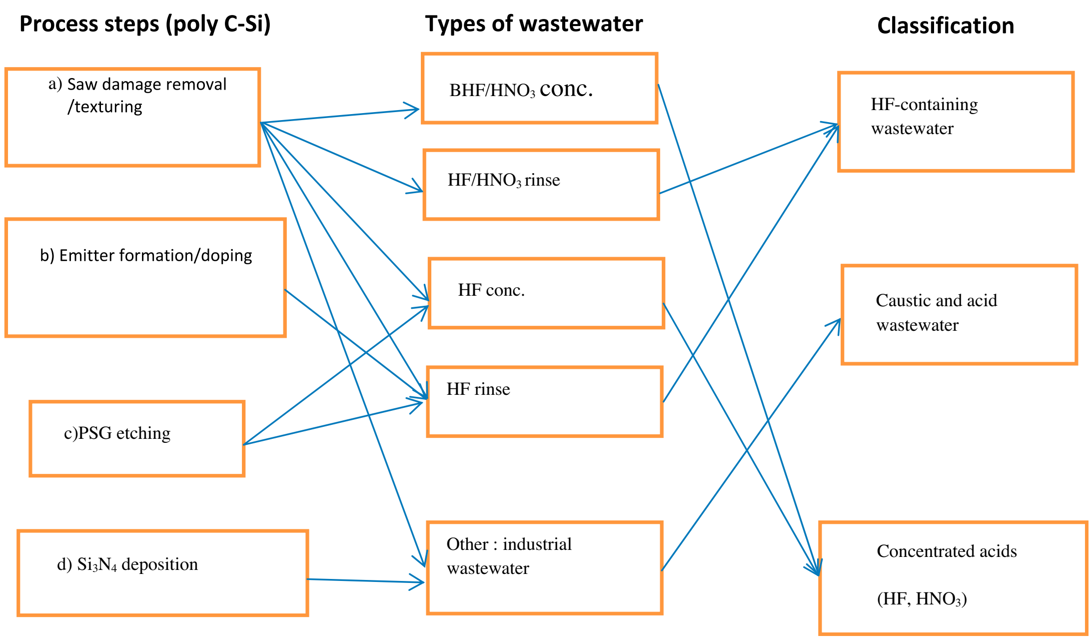

Fig. 2. Types of effluent during photovoltaic cells manufacturing [1].

typical poly-crystalline silicon. The only difference between this figure and that of solar cell-based mono-crystalline silicon production resides in the steps of the development process, as explained above [1,8] (Fig. 2).

3. Treatment methods

The usual approach for wastewater treatment in solar photovoltaic cells manufacturing is:

- Treatment of HF discharges
- Neutralization
- Collection of discharges containing isopropanol (for the production of cells based on mono-crystalline silicon.)

3.1. Fluoride wastewater treatment

Discharges containing fluoride are collected and sent for treatment in a physicochemical treatment plant. In the treatment plants, three steps are performed: neutralization, sedimentation, and filtration. Insoluble solids generated during the treatment are dried and sent to a landfill for disposal, as shown in Fig. 3.

3.1.1. Precipitation

This method eliminates substances dissolved in wastewater. This is achieved by adding chemicals that react with the targeted substance to form an insoluble compound. These reactions generally have an optimum pH that must be adjusted if necessary. Lime ($Ca(OH)_2$) or calcium chloride ($CaCl_2$), are commonly used for precipitation of fluorides, sulfates, and phosphates. The basic chemical reactions are presented below.

Case of calcium hydroxide:

$$2HF + Ca(OH)_2 \rightarrow CaF_2 + 2H_2O \quad (1)$$

Case of calcium chloride:

$$2HF + CaCl_2 \rightarrow CaF_2 + 2HCl \quad (2)$$

3.1.2. Sedimentation

Sedimentation is also called settling. It is the physical separation of suspended solids in a liquid flow using gravity. Equipment used in this process involves settling tanks, clarifiers and inclined plate clarifiers,

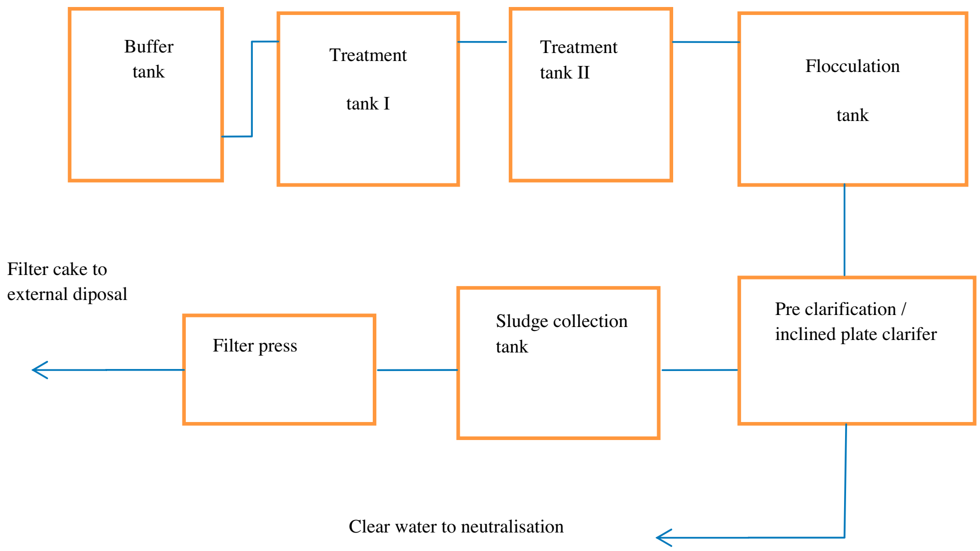

Fig. 3. Diagram of HF wastewater treatment [2].

and thickeners. Some variations in sedimentation units are inclined plate clarifiers and clarifiers. Retention time, the rate of surface load, sedimentation rate, and maintenance requirements of the reactor are the parameters that must be considered when choosing a sedimentation unit [9,10].

3.1.3. Filtration

Filtration is a separation process where a mixture of liquid and solids passes under a pressure gradient through a filter medium. The liquid passes through the filter medium as a filtrate while the impermeable substances are retained as a residue.

Various degrees of filtration can be achieved by changing characteristics of the filter; such as pore size and filter thickness. By decreasing the pore size, filter medium can vary from sand bed filter, paper, and textiles to membranes. Filtration can also be classified as frontal or as cross flow. In dead-end filtration, fluid which needs to be filtered passes perpendicular to the surface of the filter. The liquid passes through the medium as a filtrate while substances impermeable to the medium are retained as residues [10].

In the PV industry, frontal filtration (such as the filter press) is the most common approach for separating liquids and solids. Solids (as filter cake) are collected in a shipping container for land application or sent to a landfill for disposal. In most cases, the filter cake (CaF_2) is non-hazardous, but due to local requirements it may need special treatment. After HF treatment, filtrate is sent to a neutralization system for pH adjustment [10].

3.1.4. Neutralization

Fig. 4 shows a simplified diagram of a typical system of neutralization, which treats wastewater contaminated by acids or bases. This treatment consists on the wastewater pH adjustment to a neutral range between 6 and 9 or as required by regulation emission standards. To meet these requirements, the addition of acids or bases might be necessary. Sulfuric acid (H_2SO_4) and hydrochloric acid (HCl) are the most commonly used acids in this process. However, HCl generates corrosive vapors and it is not recommended for this process. H_2SO_4 is the cheapest, easiest, and safest to use. Sodium hydroxide (NaOH) is the alkaline chemical most commonly used because it is both easy to handle and inexpensive. The choice of chemicals depends on the cost and operation of the plant [10–14].

In the neutralization process, design parameters are important, for example: stirring time and dosage of reagents. These factors determine the size of the reactor and that of the metering valve. Particular attention should be paid to process control and the assay reagents [10].

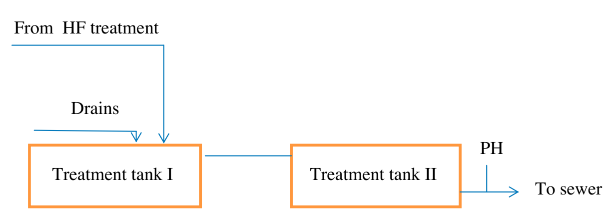

Fig. 4. Neutralization system diagram.

4. Collection of isopropanol alcohol (IPA)

In the case of solar silicon single crystal production, waste stream containing IPA is separated and collected in containers to be transported to landfill. As described above, other waste streams can be processed in a common processing system.

5. Discharges generated by silicon wafers cutting

Silicon wafers are cut with the wire saw in order to obtain wafers of 200 microns thickness. The cutting fluids are called "slurry." This is a mixture of organic solvent and abrasive form of micro beads of silicon carbide (SiC). When sawing, it takes care of the silicon particles and iron (from steel cable) [15–17].

Cutting fluids are viscous slurry consisting of solid silicon carbide and fluid glycol. Silicon carbide is used as a sawing agent in the sawing process of wafers; it is applied to the wire saw in order to provide the necessary specific hardness to saw wafers. The glycol acts as substrate as well as a coolant for the silicon carbide [18].

The effluent of cutting fluids called silicon kerf is formed as slurry along with the cutting fluid (usually polyethylene glycol) and other impurities, mainly from the broken particles of the SiC abrasive and iron from the wire. It has a non-negligible environmental and economic impact associated with the manufacturing process of PV systems. During the cutting process, they carry silicon and iron particles respectively from the base material and sawing wire. Disposal of such

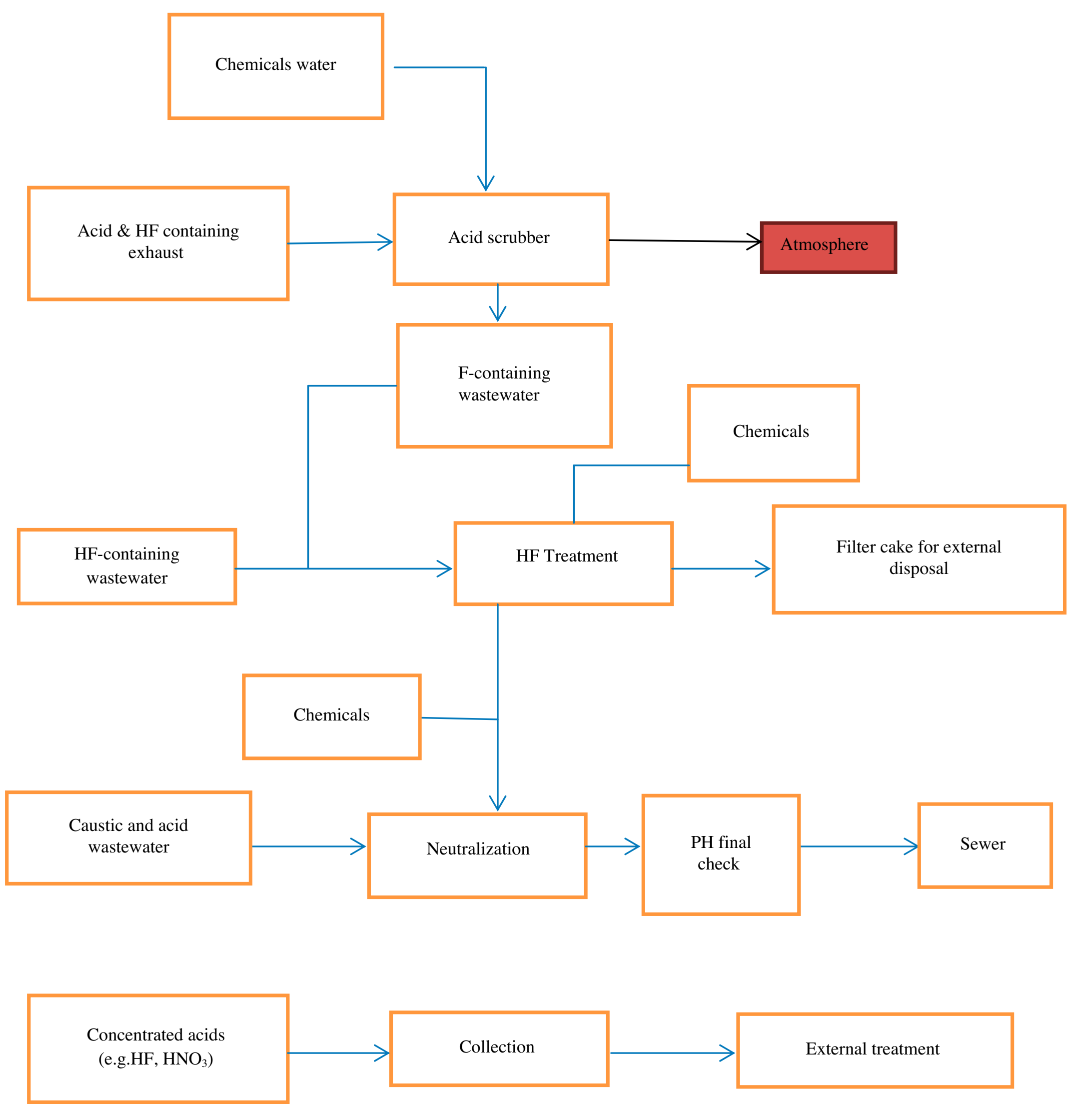

Fig. 5. Standard conception of a photovoltaic cell industry wastewater treatment plant.

waste is subject to specific regulations regarding disposal of hazardous waste, which depends on special industrial waste plans [19]. These types of wastes must not be leaved or burned in open air. Because of their highly polluting nature, it is recommended to collect, concentrate, and regenerate them [20].

Separation of the solvent and particles is possible at about 80% and the rate of reuse can exceed 80% [20]. Regeneration of cutting fluids via simple and cheap processes should allow the reuse of used fluid through recovery of its lubricating characteristics. It allows reduction of the virgin fluid consumption while preserving the environment [18].

6. Wastewater treatment plant design

Fig. 5 shows a standard installation of the wastewater treatment. In this case, the priority in terms of performance is closely related to the HF discharges neutralization treatment system.

Concentrated acids are generally collected on site in containers and then transported for external treatment. A variation of design standard for wastewater treatment does not include the HF or a concentrated acids treatment step. In this case, neutralization is the only operation used. This type of treatment system was designed and installed in practice [1].

The wastewater from scrubbers must not be neglected when designing the wastewater treatment system in terms of HF mass flow. Although the amount of water is relatively low compared to that from various production processes, it represents a significant amount in the total HF effluent flow from the manufacturing plant. This is because the wastewater generated by the scrubber depends on the type of etching process used.

In terms of discharge limits, the design of wastewater treatment plant strictly depends on its location. For example, the release limit for fluoride differs depending on the different countries or regions. Germany has no general validity. Discharge standards of fluoride depend on requirements of local authorities, ranging from less than 20 mg/L to 50 mg/L [21]. In India where natural waters are naturally concentrated in Fluor, the standard for discharge is <2 mg/L [2].

Local infrastructure is another important parameter when designing the installation of releases. Discharges of concentrated acids are usually collected and sent to a landfill or for treatment in a specialized treatment center, and if this is possible they might be treated on site. This factor must be considered when building the plant and waste treatment installation must be constructed.

7. Opportunities for reuse of rinsing water

The rinsing water of the etching process which was not contaminated with nitric acid (HNO_3) is collected separately in a receiving tank. Therefore a separate line is required to manage and to recycle only the rinsing water. In a first step, this rinsing water is filtered to prevent damage to the reverse osmosis membrane by broken particles of silicon wafers or others. The pH value of the filtered water is adjusted before the changeover to the reverse osmosis unit, where fluoride salts dissolved in the concentrate are retained [22].

This concentrate will need additional treatment to precipitate fluorides to meet specific requirements. The permeate from reverse osmosis could be recovered and used for many purposes, such as water supply for scrubbers and cooling towers [1,22].

8. Zero discharge concepts

The production of solar cells is increasingly transferred to countries in which the cells produced can be effectively used on account of intensive solar radiation. In these countries (in southern Europe or northern Africa, for example), there is often a severe shortage of water. For this reason, concepts for water recycling, going as far as "zero discharge" are sensible and cost-effective there.

Such a concept has been devised for a customer. From river water treatment to recirculation, a complete process for water management with "zero discharge" criteria was developed. The important thing is to master the production process in order to be able to close the circuit. The production in question is a wafer and cell production system (Fig. 6). The wastewater is classified according to the following criteria:

- Sanitary wastewater from the administration department.
- Organically contaminated rinsing water from wafer production.
- Inorganically contaminated concentrates from cell production.
- Inorganically contaminated rinsing water.

The weakly contaminated rinsing water is treated by reverse osmosis process after an appropriate conditioning. Permeate is fed back before the water treatment plant for high-purity production water. This makes it possible to save considerable quantities of water.

The concentrates from the reverse osmosis as well as for all other wastewater are treated in the chemical–physical treatment plant. Uniform inflow

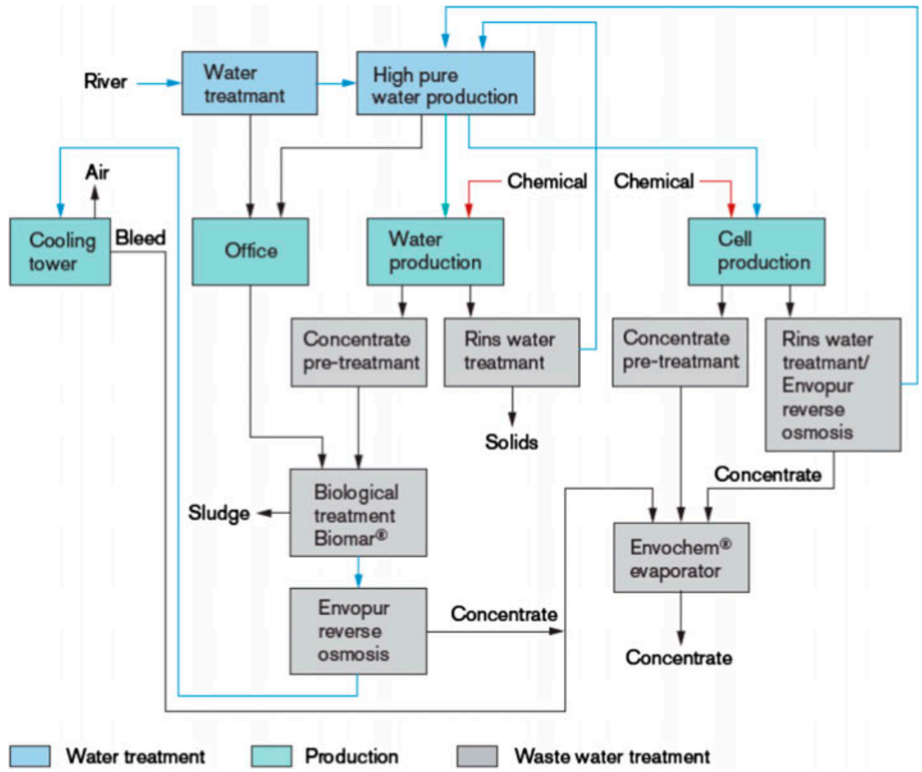

Fig. 6. Complete process for water management [22].

conditions are important for stable functioning. For this reason, concentrates (discontinuously discharged or rejected treatment baths) are collected separately and then dosed. The pretreated inorganic wastewater is then evaporated. All organically contaminated wastewater is subjected to anaerobic biological treatment. The cleaned wastewater is treated by membrane technology in order to be used in the cooling tower [2].

9. Conclusion

Photovoltaic (PV) energy conversion is increasingly regarded as a technology which may contribute to the world energy supply in a way that is compatible with the concept of sustainable development. However, to ensure that PV energy can indeed fulfill this expectation, a careful consideration of potential environmental risks of PV energy conversion is necessary. PV manufacturing process, which requires extremely high precision, generates both conventional and hazardous wastes.

The management of waste has become an important issue in the industry as a result of stringent environmental regulation and possible liability. Among varieties of pollutants, hydrofluoric acid (HF) is a major concern. It is used extensively in PV manufacturing for wafer etching and quartz cleaning operations. Fluoride concentrations of 500–2,000 mg/L are found in typical wastewater of local PV industry. Fluoride contamination in certain aquatic systems worldwide has caused health concern. Due to its high toxicity, industrial wastewater containing fluoride is strictly regulated.

Contamination can be safely and efficiently removed from wastewater using conventional precipitation/neutralization processes or by especially engineered polymers and membrane technologies to comply with the strictest standards. Using the new treatment technology, photovoltaic cell industries can reduce operating costs, reprocess the water to allow either recycling within the same processes or reuse it in another process in the facility, comply with expanding environmental regulation and improve relations within the local community.

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