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Recovery of valuable products from kerf slurry waste – case of photovoltaic industry

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ABSTRACT

Although solar cells manufacturing is a conservative industry, economics drivers continue to encourage innovation, feedstock savings and cost reduction. Kerf slurry waste is a complex product containing both valuable substances as well as contaminants. The valuable substances are: (i) high-purity silicon, (ii) polyethylene glycol, and (iii) silicon carbide. The contaminants mainly include metal fragments and organics. Therefore, recycling of the kerf slurry waste is an important subject not only from the treatment of waste but also from the recovery of valuable products. The present paper relates to processes for the recovery of valuable products from the kerf slurry waste in which they are contained, such products comprising nanoparticles, polyethylene glycol, high-purity silicon, and silicon carbide.

Keywords: Kerf slurry waste; High-purity silicon; Polyethylene glycol; Silicon carbide; Photovoltaic

1. Introduction

The renewable energy is considered part of the future energy sources and plays an important role in the present and future energy scenarios. Nowadays, the photovoltaic solar energy is a practical and economical alternative instead of the energy produced by fuels for a wide variety of applications. Over the last decade, recycling of high-purity silicon has become an important issue for the solar cell production industry due to the lack of sufficient feedstock supply, the high price of silicon, and the high production cost. More than 40% waste is derived from the slicing process or ingot top-cut scraps and the kerf loss or slurry waste [1–3].

The recycling or recovery of silicon as valuable material has gained a significant interest from many researches [4]. It not only represents an environmental friendly manner of managing the production of wastes but also represents a great challenge and extensive research [5]. Proper recycling of kerf slurry waste ensures that potentially harmful materials are not released into the environment, and reduces the need for silicon solar-grade material. An example is the recovery of pure silicon from damaged or end-of-life Photovoltaic (PV) modules, which leads to environmental and economic benefits. Moreover, the recycling of slurry waste is able to add value mainly in terms of financial, payback time and energy consumption, as well as CO_2 emissions reduction. The manufacture of a silicon ingot for photovoltaic applications is a very energy-intensive process with high production costs [6–8]. In contrast, the financial cost for recycling kerf slurry waste could only represent ~5% of the primary production of solar-grade silicon. Furthermore, energy payback is one of the main issues for the silicon photovoltaic industry. It is assessed by the cost per watt of

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power generated and the energy generated when the solar cell exceeds the energy consumed by its production. Recent studies indicate that photovoltaic silicon systems currently have an energy payback time of 1.5–3.5 years for locations with irradiation conditions of 1,000–1,700 kWh/m^2/yr. In recognition of these facts, the photovoltaic industry is acting voluntarily to implement product take-back and recycling pathways at the manufacturing level [9,10]. Depending on the technology, the kerf slurry waste can then either be recycled to serve as feedstock for new polysilicon solar cell or be recovered valuable substance as polyethylene glycol or silicon carbide (SiC) [11]. The present paper is a review of the most significant and updated information on innovative processes for silicon slurry waste valorization such as physical and chemical separation, centrifugation, aerosol-assisted spray-drying extraction, and membrane technique.

2. Kerf slurry waste

Multi-wire slicing technology has been widely used in manufacturing of silicon wafer intended for solar cells. Solar-grade crystalline silicon materials (SoG-Si) are used as the substrate in solar cells production. A multi-wire saw with polyethylene glycol, containing SiC abrasives, is used as vehicle during the processing.

Wafer production starts by cropping and followed by bricking. Subsequently, the wafering operation is typically performed by means of wire saws. The ingot is contacted with the alternative wire while the liquid slurry, containing SiC grains and polyethylene glycol, is supplied to a contact area between the ingot and the wire saw. During silicon wafer production for solar cells, a great amount of kerf loss silicon is produced. The kerf slurry waste is a by-product derived from sawing silicon ingot into wafers. It is composed of pure fine particles of silicon, polyethylene glycol solution, abrasive SiC particles, and metal impurities.

3. Pathways for kerf slurry waste valorization

3.1. Liquid–liquid extraction method

The liquid–liquid extraction method consists in stirring sufficiently a water-particle-oil system. Hydrophobic particles will not stay in the water phase but go into the oil phase; the opposite happens to the hydrophilic particles. When the oil and water de-mix, the separation of the particles is complete.

Liquid–liquid extraction when using wet oxidation has been found to be an effective method for Si recovery from the slurry waste. The experiment consisted in using kerosene as the oil and pure water. The slurry was a wet oxidized artificial Si-SiC powder mixture. Nitric acid was used as oxidant agent to the slurry powders before liquid–liquid extraction. The slurry was first mixed with pure water under constant agitation. After that, kerosene and more pure water were added to the mixture. Then the solid–liquid system was allowed to settle until the oil and water phases were de-mixed. These phases contain SiC and Si particles, respectively. Most SiC particles go to the oil phase while Si particles accumulate at the bottom in the water phase (Fig. 1). As a final step, the powders from each phase are collected and washed before drying at 60°C. From this experiment, a fraction value of 0.034 of Si consumed in oxidation resulted in a Si recovery with high purity. Therefore, an optimal degree of oxidation is an important factor for this technique [12].

Fig. 1. A picture of phases in the separating funnel after settling for 10 min during recovering Si from wire saw slurry by liquid–liquid extraction, with kerosene as the oil. Slurry is wet oxidized, and {O} content is 1.7% by weight.

3.2. Development of recovery system for extracting silicon carbide from photovoltaic industry abrasive slurry

SiC recovery is of high interest for the photovoltaic industry. This ceramic material is extensively used as an abrasive material for sawing silicon ingot into individual wafers.

Yang et al. [13] developed a pilot-scale physical and chemical separation system for the recovery of SiC from the abrasive slurry derived after the cutting, grinding, and polishing process (Fig. 2). The system was a recycling technique that removes magnetic materials (metals) and silicon particles dispersed in the aqueous slurry waste by adding sodium hydroxide and sodium metasilicate. Subsequently, the SiC was recovered by dispensing the liquid and drying the solid slurry in a rotary kiln.

After the separation process, a high-purity fine powder consisting only of SiC and no residual silicon was satisfactorily obtained.

3.3. Hydrogen production

Using the centrifugation method, the SiC and the cutting fluid was separated from a sample of silicon slurry waste derived from wafer slicing (Fig. 3). After removing the organic impurities from the sample, a so-called “original powder” (69.3 wt% Si, 13.2 wt% SiC, 17.5 wt% metals) was obtained.

Hydrogen from the original powder was produced by chemical reaction between Si and H_2O using as catalyst alkali. Applying ultrasonic treatment, the powder mixed with deionized water was well distributed. Subsequently,

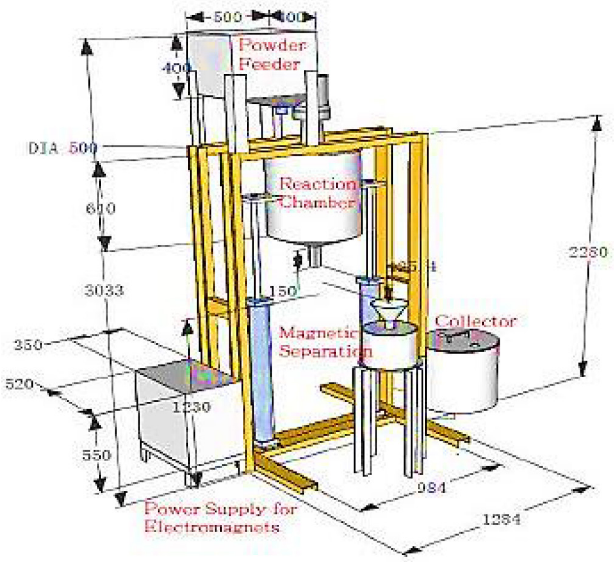

Fig. 2. Scheme of pilot-scale recovery system.

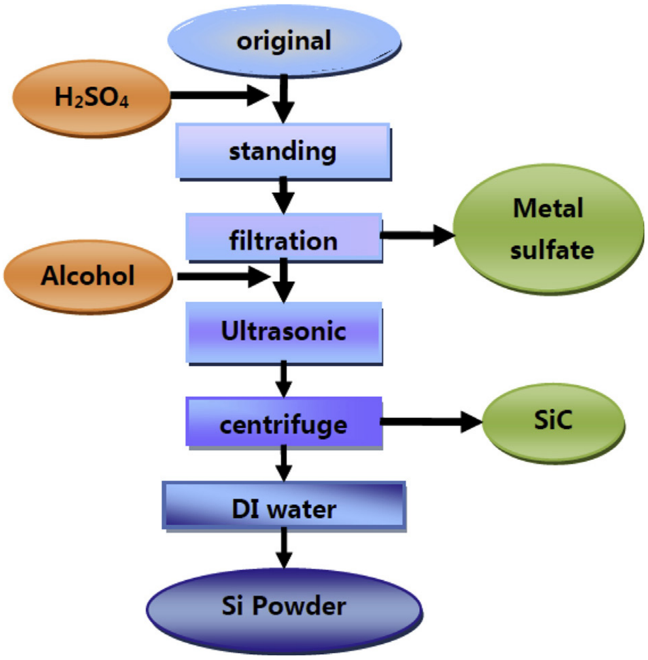

Fig. 3. Flowchart for the removal of metals and considerably larger sized SiC.

sodium hydroxide and sodium silicate played the role of reaction promoters during the transition state of the chemical reaction. The fluid sample stirred using a magnetic stirrer. The amount of gas was determined via gas chromatography.

Throughout this experiment, hydrogen from silicon powders was also produced using irradiation method. Under light irradiation, the hydrogen generation may start by photoexcitation of the powder and chemical reactions [14].

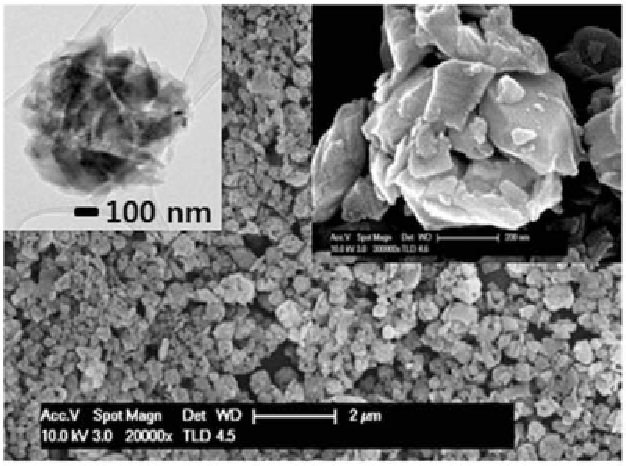

Fig. 4. Scanning electron micrograph of the silicon nanoparticles obtained after aerosol extraction.

3.4. Nanoparticles

3.4.1. Aerosol-assisted extraction of silicon nanoparticles from wafer slicing waste for lithium ion batteries

As a one-step separation process, the aerosol-assisted spray-drying extraction is a suitable and low-cost method to recover the silicon nanoparticles for the lithium ion batteries manufacturing.

The Si and SiC debris are dispersed in water and subjected to an ultrasonic atomization in order to disperse the different particles and nebulize the water droplets, while the Si nanoparticles are ground into small Si particles by the bigger SiC particles. The introduction of the Ar gas leads to a flight of the water droplets in the preheated tube furnace, while the evaporation of the solvent generates a sufficient capillary force to assemble the Si nanoparticles into clusters. Aerosol extraction allowed 80 wt% of Si recovery from the silicon sludge, whereas only 3.8 wt% of SiC are recovered.

The Si nanoparticles recovered are not as uniform in size and shape as available in the market (Fig. 4). However, the recovered silicon nanoparticles agglomerates have better retention capacity than commercially Si nanoparticles when used as solid anode in lithium batteries [15].

3.4.2. Scalable upcycling silicon from waste slicing sludge for high-performance lithium-ion battery anodes

The wrapping of Si nanoparticles with the multi-layers of graphene is achieved by the combination of a spray-drying method and high-energy ball milling process, and exhibits a promising performance as Lithium ion batteries (Libs) anodes. The Si nanoparticles are recovered from waste slicing sludge using a spray-drying method. It consists on a passage of the silicon sludge from a rotary atomizer into a vertical co-current drying chamber under a compressed air atmosphere.

The second step is the pulverization of the recovered silicon using a high-energy agate ball milling. The recovered silicon is put in a grinding vial at 900 rpm with a 20:1 weight ratio of agate balls to silicon powder. After 5 min of milling, the grinding is broken for 45 min, and both graphene oxide (20 wt%) and cationic polyelectrolyte (10 wt%, PAH, Sigma-Aldrich-FRANCE) are added. This mixture is then subjected to a second high-energy ball milling for 2 h.

Scanning electron microscopy (SEM) and Ultra-high resolution transmission electron microscopy (HR TEM) images show the nanometer sized of as milled silicon (10–150 nm) and the graphene shell well wrapped silicon nanoparticles, respectively (Fig. 5).

Cyclic voltammetry, electrochemical impedance, and charge/discharge cycling life measurement confirmed a successful recovery of silicon waste into a high-performance Libs anode [16].

3.4.3. Preparation of silica nanoparticles from waste silicon sludge

The dissolution-precipitation technique optimized by a response surface methodology (RSM) is used to recover the silica from the waste silicon sludge (WSS). This approach consists in adding WSS into a dissolution of the sodium hydroxide (NaOH) in 20 ml deionized water. After that, the resultant slurry is cooled to room temperature. The SiC is filtered off, and the sodium silicate is stirred and titrated (with 20% HCl at 80°C) until the silica gel precipitates.

Prior to initiating the experiments, a statistical tool is used to optimize the silica dissolution parameters. The optimized conditions are related to the mass ratio of NaOH and WSS, the temperature, and the experiment duration.

The predicted efficiency of the technique, estimated by 15 runs of Box–Behnken design (BBD), is 97.27% under the following optimum conditions: 1.73 mass ratio NaOH/WSS, 118.8°C, and 3 h experiment duration. In fact, the efficiency value resulting from different experiments under these conditions confirmed the predicted efficiency.

The silica nanoparticles viewed by TEM exhibit a 20–45 nm diameter (Fig. 6) and appeared as high pure amorphous silica when analyzed by ray diffraction (XRD). This process confirmed a high suitability of an efficient dissolution-precipitation of silica [17].

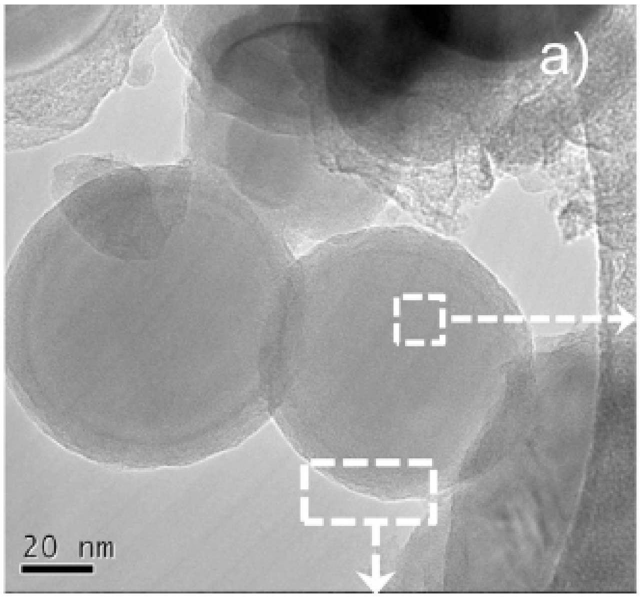

Fig. 5. Transmission electron micrograph of graphene encapsulated silicon nanocomposites.

3.5. Polyethylene glycol recovery

3.5.1. Chemical pretreatment and ultrafiltration

Important quantities of silicon and fluid cutting are wasted in sawing operation. This waste leads to an increase of economic loss, photovoltaic energy cost, and environmental pollution. The usual methods for waste treatment achieve a low efficiency. As a result, a simple low cost and efficient method has been investigated in order to regenerate a cutting fluid.

The cutting fluid provided by the Unit of Silicon Technology Development of Algiers (UDTS) is chemically pretreated using an industrial quality of dichloromethane. A proportion of 10% of this organic solvent is mixed with cutting fluid and stirred for 1 min. After settling, the resulting liquid is ultrafiltrated using a polyethersulfone membrane of 1 Kg Da.

Via the representation of the permeate flux vs. the time of the ultrafiltration process at a pressure of 1 bar, it is easy to notice that the flow of pure fluid cutting is constant while the flow of the used fluid cutting decreases rapidly in permeate flux. The variation of the permeation flux of the chemically pretreated fluid is very comparable with the one of the pure fluid cutting. Fourier transform infrared spectroscopy (FTIR) analysis confirms clear similitudes in chemical bonds between the chemically pretreated-ultrafiltrated fluid and the pure fluid cutting (Fig. 7).

This work allowed proving the potential regeneration efficiency of the ultrafiltration of the chemically pretreated cutting fluid [11].

3.5.2. Centrifugation

A great amount of slurry waste is formed during the cutting process from silicon ingot to wafers. More than 50% of this waste comes from cutting liquids. Regeneration and recovery of cutting fluids via simple and cheap processes should allow the reuse of used fluid, and might substantially reduce the amount of silicon cutting waste

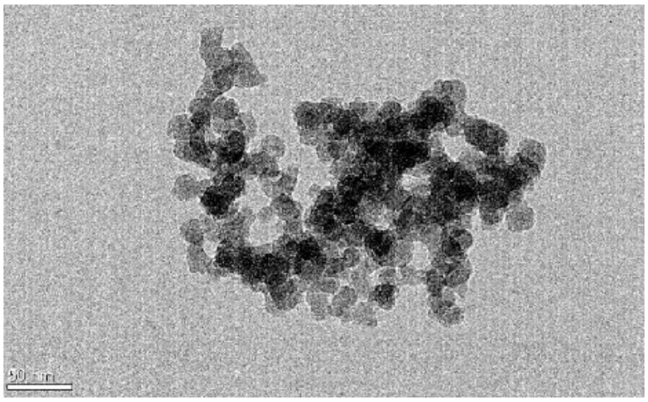

Fig. 6. Transmission electron micrograph of silica nanoparticles recovered from WSS by the dissolution-precipitation technique.

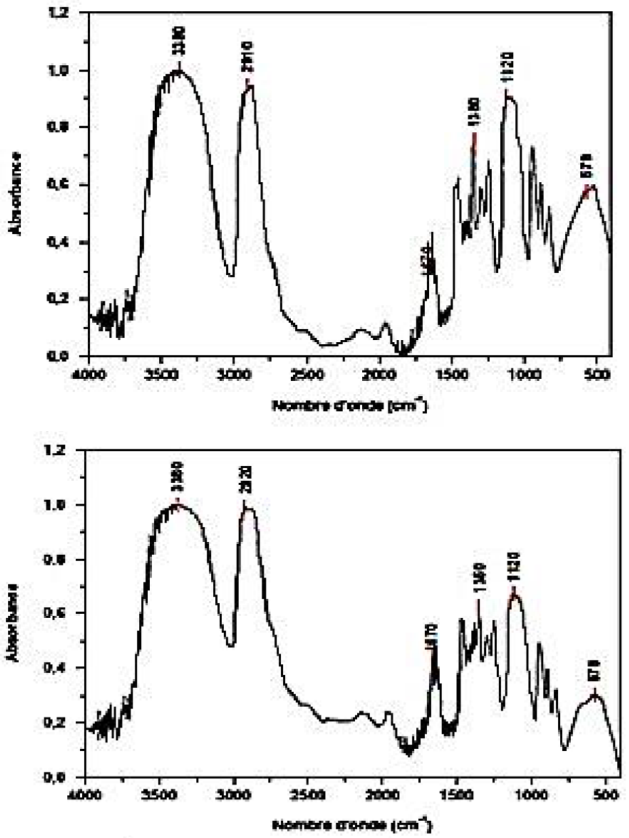

Fig. 7. FTIR spectrum of: (a) the regenerated fluid and (b) the pure fluid.

and photovoltaic industry wastewater with high chemical oxygen demand, which is difficult to treat and becomes a considerable environmental issue [18–20]. Tsai et al. [21] investigated centrifugation technique to recover polyalkylene glycol (PAG) cutting liquid with acceptable final turbidity (<100 NTU) (Fig. 8). They demonstrated that clean PAG liquids could be obtained by using water as a diluent. To obtain a low-turbidity PAG liquid, less than 100 NTU, from silicon cutting waste, the authors adjusted the operational variables of centrifugation and employed acetone, ethanol, and deionized water as diluents for enhancing separation performance. With 50% (wt), water-assisted centrifugation at 3,253 G-force for 24 h, the solid content of the liquid decreased to 0.018 g/L, and the turbidity reduced to 7.2 NTU. The obtained liquid was then vacuum distillated to remove water. Thus, the recovered PAG with only 0.43 NTU could be reused in the cutting process with glycol recovery percentage of about 95%. The authors concluded that recycling this glycol liquid from cutting waste would greatly reduce the amount of waste, and thus, the total slicing cost can be efficiently reduced.

3.6. Cementitious material

Solid waste is sludge composed of agglomerates of nanoparticles like SiO_2 and $CaCO_3$ generated from photovoltaic-industries wastewater. This sludge deflocculates in aqueous solutions into nanoparticles smaller than 1 µm. Thus, this sludge is hazardous when it is improperly disposed but due to its high content of amorphous SiO_2, this sludge has a potential use as supplementary cementitious material (SCM) in concrete. Quercia et al. [22] demonstrated that the silica-rich waste sludge (nSS) can be utilized as SCM to replace the portion of cement in mortars, thereby decreasing the CO_2 footprint and the environmental impact of concrete. In order to assess the potential use of nSS as SCM, the main properties of several photovoltaic's silica-rich sludges were physically and chemically characterized. The main characterization techniques were: SEM, X-ray energy-dispersive spectroscopy (EDS), XRD, nitrogen physical adsorption isotherm (Brunauer-Emmett-Teller BET theory), density by helium pycnometry, particle size distribution determined by laser light scattering (LLS), and zeta-potential measurements by dynamic light scattering (DLS). In addition, a dispersability study was performed in order to design stable slurries to be used as liquid additives for the concrete production in site. The effects on the hydration kinetics of cement pastes by the incorporation of nSS in the designed slurries were determined using an isothermal calorimeter. A compressive strength test of standard mortars with 7% of cement replacement was performed to determine the pozzolanic activity of the waste nano-silica sludge. Finally, the hardened system was fully characterized, so that the phase composition could be determined.

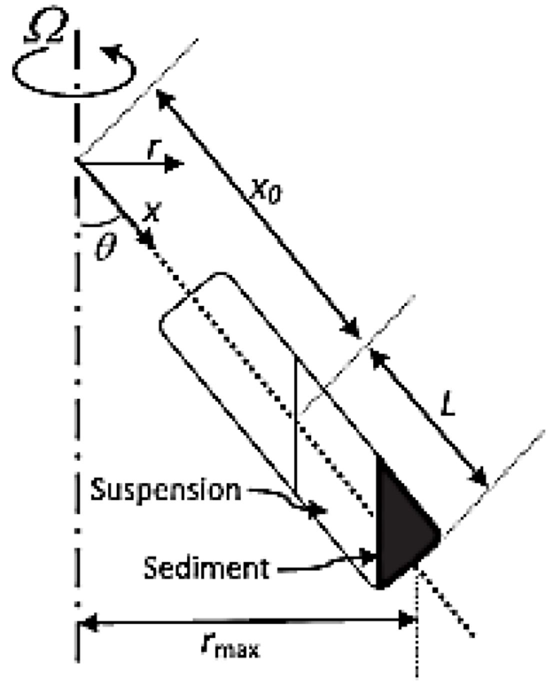

Fig. 8. Schematic diagram of the centrifuge.

Quercia et al. [22] mainly concluded that the chemical analyses revealed a high content of amorphous SiO_2 and $CaCO_3$ with some impurities related to the additives used to prepare the original polishing slurries. And, that the resulting slurries were stable and easy to prepare for the application in concrete.

The detailed characterization of the hydrated system with photovoltaic's silica-rich waste demonstrated that the replacement of cement with this material influenced the amount and kind of formed hydration phases and thus the volume, the porosity, and finally the durability of the tested mortars (Fig. 9).

3.7. Silicon carbide recovery

The increasing demand for silicon solar cells has increased the amount of produced silicon sawing waste. Recycling kerf Si and SiC from sawing waste is an economical method to reduce the solar silicon sawing waste. However, the separation of Si and SiC is a difficult task.

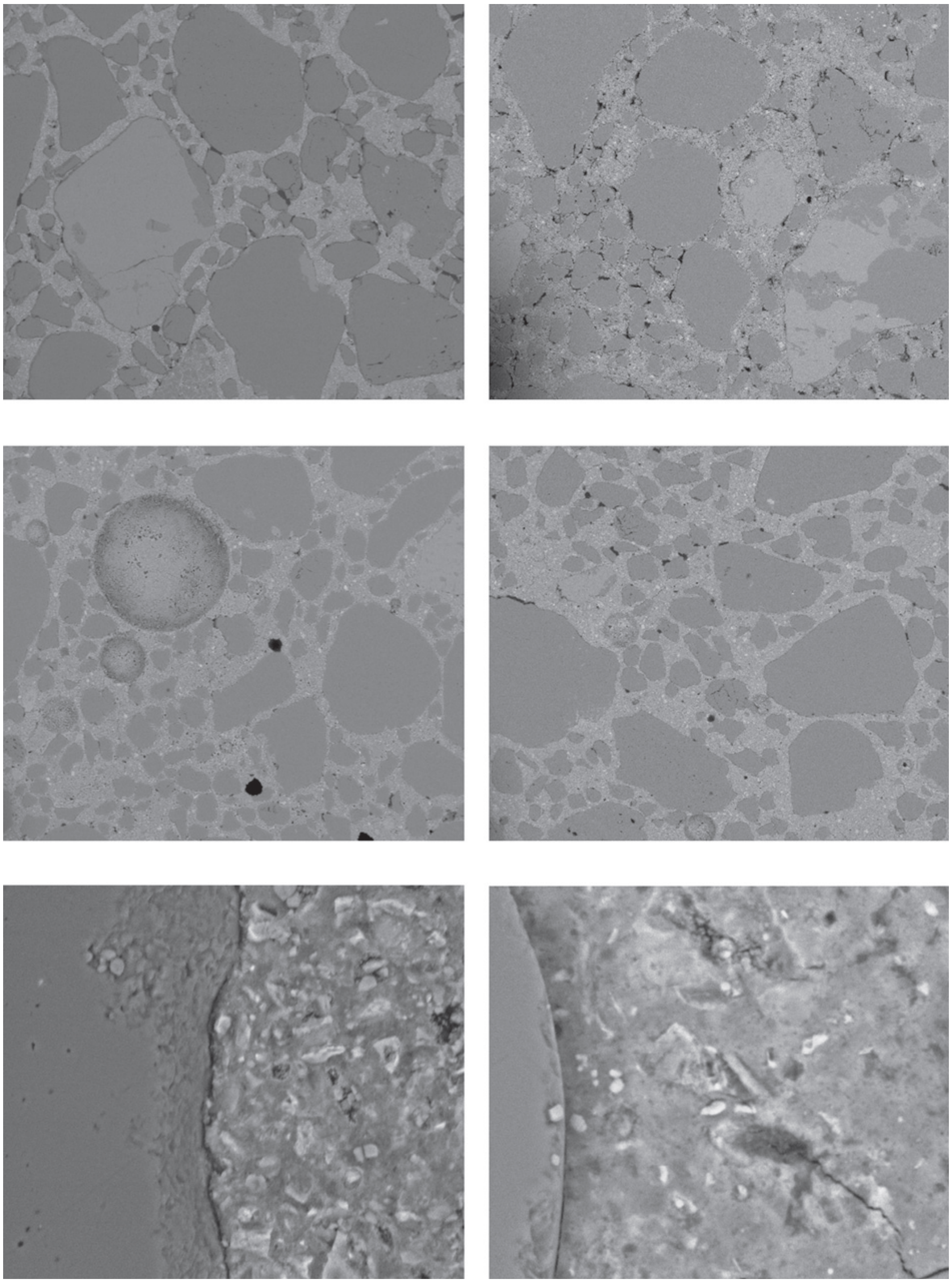

Fig. 9. Environmental scanning electron microscope (ESEM) with efficient backscattered electron detectors (BSE) pictures of different mortars tested at 28 d: (a) reference, (b) batch 2, (c) batch 1, (d) micro-silica, (e) detailed interfacial transition zone (ITZ) in batch 1 mortar and (f) detailed ITZ in micro-silica mortar.

Tsai et al. [23] studied the separation of Si and SiC using a ramp settling tank. They found that with a settled electrical field, small Si particles with higher negative charges have a longer horizontal displacement than SiC particles in a solution with a pH value of 7, resulting in the separation of Si and SiC. The agreement between experimental results and predicted results showed that the particles traveled a short distance to reach the collection port in the ramp tank. Consequently, the time required for tiny particles to hit the tank bottom decreased, and the interference caused by the dispersion between particles and the fluid motion during settling also decreased. In the ramp tank, the highest purities of the collected SiC and Si powders were 95.2 and 7.01 wt%, respectively. In addition, the recycling fraction of Si-rich powders (SiC < 15 wt%) reached 22.67%, which is greater than that achieved using previous rectangular tanks.

The proposed process by Tsai et al. [23] (Fig. 10) consisted in mixing and oscillating in a shaker for centrifugation, an amount of sawing waste with nitric acid to dissolve metal impurities such as iron, copper, and zinc. The remaining wet cake was then washed with acetone and centrifuged in order to remove liquid components. The cake was later dried in an oven to produce solid powder. The solid components were 65–71 wt% of the original sawing waste. After that, the authors suggested a separation process in which a quantity of the obtained solid powder was mixed with the neutral (pH 7) solution (prepared from phosphoric acid and sodium hydroxide) and transferred to a settling tank (Fig. 9). A basic sedimentation cell was used to separate different particles under gravity and electrical fields. A metering pump at the cell inlet inputs the solution containing SiC and Si particles. The flow rate was just low enough to overflow the barrier between the test solution reservoir and the inlet of the settling tank. A power supply and a pair of platinized titanium electrodes created a constant electrical field across the solution in the tank. The tank bottom was equally divided into 10 regions to collect the settling particles. After settling for 24 h, the refined solid was collected.

In conclusion, compared with the rectangular tanks, the recycling fraction of Si-rich powders using a ramp tank is greater. Also, the ramp settling tank proposed by Tsai et al. [23] is more suitable for industrial applications. The Si recovered near the outlet can be refined further by using the high-temperature treatment and the dimensional solidification method to produce solar-grade Si.

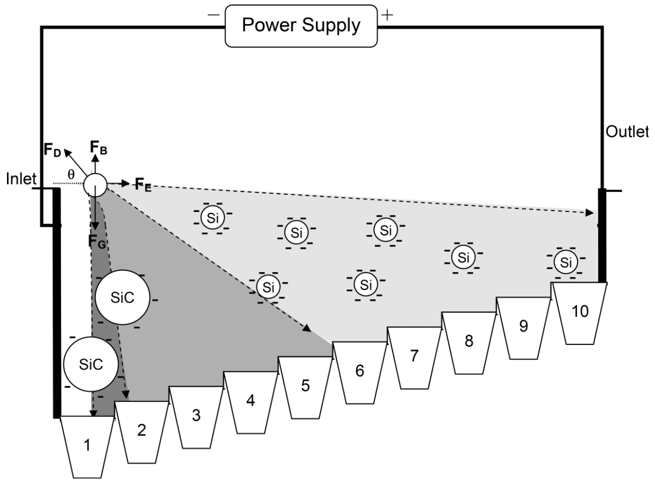

Fig. 10. Schematic diagram of particles settling in a ramp tank under an applied electrical field.

4. Conclusion

Comprehensive review of literature indicated that the recovery of valuable by-product from kerf slurry waste requires an integrated systematic approach. Since the recovery of each of the different compounds from slurry waste requires very specific techniques and/or methods, the understanding of the suitable process influencing compound separation was emphasized. Research and development of innovative and feasible approaches for recovering low-cost by-products from kerf slurry waste are ongoing. They represent a need for the photovoltaic industry in the present and in the near future.

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