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Recycled polycrystalline CdS film electrodes with enhanced photo-electrochemical characteristics

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ABSTRACT

Waste cadmium sulfide (CdS) film electrodes, originally deposited onto glass/fluorine doped tin oxide (glass/FTO) substrates, were used to prepare recycled CdS film electrodes. The waste glass/FTO/CdS were processed in acidic media to recover the glass/FTO substrates, the Cd^{2+} ions (in the acidic solutions) and the gaseous H_2S (recaptured in basic media). All components of the waste electrodes were thus recovered. The recovered glass/FTO and the Cd^{2+} ions were then reused to produce new recycled glass/FTO/CdS electrodes by chemical bath deposition. The produced films were then characterized by X-ray diffractometry, scanning electron microscopy, electronic absorption spectroscopy and other techniques. The Cd^{2+} ions were recovered with efficiency higher than 90% from the waste films, as observed from atomic absorption spectrometry. The recycled films were assessed in photo-electrochemical conversion of light to electricity, and exhibited comparable efficiency to those freshly prepared from authentic starting materials and other literature values. Photoelectrochemical characteristics for the recovered films were further enhanced by avoiding stirring of the chemical deposition bath during preparation. The results manifest the feasibility of recycling CdS electrodes and enhancing their photo-electrochemical characteristics by simple low cost methods. Both environmental protection and economic goals can thus be potentially achieved.

1. Introduction

As photo-voltaic PV systems involve advanced preparation techniques, and demand huge amounts of starting materials to prepare relatively thick wafers (mm scale thickness), attention is now paid to other less demanding alternative photo-electrochemical (PEC) processes. Currently research is active on PEC processes which use thin film electrodes, for light-to-electricity conversion. The film electrodes typically involve nano-size polycrystalline materials, most commonly metal chalcogenides (MX: M = Cu, Zn, In, Cd, Sn; X = S, Se, Te). Basic and applied research involves preparation methods, composite materials, efficiency & stability enhancement, characterization, and other optimization study. Both theoretical, modeling and experimental studies are active in these areas.

Due to the wide application of PV systems which involve homo- and hetero-junctions of Si, GaAs, InP, in addition to many other elements (such as Ge, Se, Te, Cd), such materials are now becoming a cause of concern to the environment. Many elements involved in PV systems are considered to be environmentally hazardous. Efforts are now being made to recover different elements from waste PV grids [1,2].

Film PEC systems have not yet reached the wide application of PV systems, and are still more at the research level. Naturally research is focused on their efficiency more than on their environmental impact and recycling. Therefore, less attention has been paid to recycle hazardous elements commonly used in PEC film electrodes. Malinowska et al. reported how CBD deposited CdS electrodes can be recovered in terms of Cd^{2+} ions and how the reclaimed ions can be reused to deposit new CdS films by a number of methods [3]. Other methods have been proposed to make benefit from waste CdS and other photovoltaic materials [4].

CdS electrodes are intentionally chosen here for more than one reason. CdS films are being widely studied in PEC research. PV systems using p-n junctions of Cu_2S/CdS were reported as early as 1954 [5]. CdS is used commercially in yellow dyes [6,7], solid state lasers [8] and

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other applications [9]. Moreover, Cd ions are highly hazardous and are believed to be carcinogenic. They are slowly absorbed by the body, but once absorbed they cause troubles to the kidneys and the lungs [10]. The US EPA recommended maximum concentration limit for Cd^{2+} ions in drinking water is 0.005 mg/L (5 ppm) [11–13]. For these reasons, it is necessary to find simple and practical strategies to prevent dumping Cd ions to the environment and to reclaim any traces present in the environment. Recycling waste CdS electrodes commonly used in PEC processes could be a viable method to achieve these strategies.

Different techniques have been used for this purpose, including ion exchange, coagulation, chemical precipitation and others. Precipitation has been widely used for Cd recovery [14]. In precipitation, the Cd^{2+} ions are allowed to react with an anion to precipitate. Carbonate [15], hydroxide [16] and other ions are useful for this purpose. Depending on the used pH value, Cd^{2+} ions can be lowered from ~ 7000 mg/L to as low as 0.3 mg/L using precipitation with carbonate [15]. Hydroxide precipitation studies showed that up to ~ 99% removal can be achieved with hydroxide, starting with 100 mg/L going down to less than 0.3 mg/L at pH 9 [17]. The sulfide precipitation has been reported to lower the Cd^{2+} ion concentrations in aqueous solutions [18]. Each of these methods has its own advantages and disadvantages, while the sulfide precipitation seems to have the upper hand due to many reasons [14].

In this work, more than one objective is targeted. Recovering residual Cd^{2+} ions from waste CdS film electrodes will be assessed. The glass/FTO substrates will be recovered, and the sulfide ions will also be recovered. Preparing new recycled CdS film electrodes will then be deposited onto the recovered substrates. The recycled CdS film electrodes will then be assessed in PEC processes. Unlike earlier reports, the recycled CdS electrodes will be characterized and tested for real light-to-electricity conversion, in comparison with other authentic CdS electrodes. Enhancing the recycled electrode conversion efficiency will also be assessed.

2. Experimental

2.1. Materials

Common solvents and chemicals were purchased from Aldrich, Frutarom and Merck. CdS films prepared and used in earlier research activities were used for recycling purposes here. The deposition procedure for the original waste films is described in earlier reports [19,20]. The films were earlier deposited onto glass/fluorine doped tin oxide (glass/FTO) substrates (Aldrich, with surface resistivity ~ 7 Ω/sq).

2.2. Equipment

Solid state electronic absorption spectra were measured for CdS films on a Shimadzu UV-1601spectrometer. Glass/FTO substrates were used for baseline correction. A Perkin-Elmer LS 50 Luminescence Spectrometer was used to measure the film photoluminescence spectra using 390 nm wavelength for excitation, and a cut-off filter (removing 450 nm and shorter).

X-ray diffraction (XRD) patterns were measured on an XRD-7000, SHIMADZU X-ray diffractometer, with a CuKα (λ = 1.5406 Å) source at ISAA Environment Consulting Co. Ltd, Chungju City, S. Korea. Scanning electronic microscopic (SEM) micrographs and energy dispersive X-ray spectroscopy (EDX) were measured on a Field Emission Scanning Electron Microscope FE-SEM, JEOL JSM-6700F at same place. Additional XRD and SEM measurements were made, for confirmation, at Pukyong National University, 365 Sinseonro, Namgu, 608739 Busan, South Korea.

Cd^{2+} ion concentrations were measured using atomic absorption spectroscopy (AAS). A Thermo 50–60 Hz Type ICE 3500 atomic absorption spectrometer was used for this purpose. Pre-calibration was made using known Cd^{2+} ion concentrations. Illumination was performed using a 50 W halogen spot lamp, with wide spectral range 450–800 nm and high stability. A Lutron-LX 102 lightmeter and a Zonen CM11 Pyranometer were used to measure light intensity.

2.3. CdS film recycling

Waste CdS film electrodes used in earlier studies were immersed in minimal amounts of HCl solutions with different concentrations (0.6, 0.8 and 1.0 M) for 5 min. The 1.0 M concentration was more suitable and was used unless otherwise stated. The acidic solution was then taken with the Cd^{2+} ions for further use. AAS was used to measure Cd^{2+} ion concentrations in recovered solutions. The resulting H_2S gas was channeled through tubes and traps and captured separately inside NaOH (1.0 M) solution. Depending on number of waste CdS film electrodes added and amount of HCl used, different Cd^{2+} concentrations were recovered. For practical purposes, enough waste electrodes were added so as to reclaim a solution with 0.2 M, for recycled electrode purposes. Higher than 90% of Cd^{2+} ions could be recovered from the waste.

The waste glass/FTO substrates (originally purchased from Aldrich as highly transparent conducting slides) were then taken from the acidic media and washed with distilled water. The substrates were cleaned by standard methods for re-deposition purposes, by treatment with concentrated HCl (1.0 M) for 60 min followed by methanol for 30 min in a sonicator. The substrates were then immersed again in dilute HCl (10% v/v) for 5 s, rinsed with distilled water, immersed in methanol and rinsed with distilled water. The cleaned glass/FTO substrates were used for recycled CdS electrode preparations, while fresh substrates were used to prepare fresh glass/FTO electrode after precleaning as described above.

The recovered Cd^{2+} solutions were then used to deposit recycled CdS films onto the recovered Glass/FTO substrates by chemical bath deposition (CBD) method as described earlier [21–24]. Distilled water (25 mL) was added to 2.5 mL of recycled Cd^{2+} solution (0.20). To the solution were added solutions of NH_4Cl (10 mL, 0.20 M) and NH_4OH (15.0 mL, 2.0 M), to make the overall solution basic. Preparation batches were made with or without stirring, for further enhancement as described below. This was to check effect of stirring on recycled CdS electrodes. The temperature was kept at 80 °C during the deposition process. The glass/FTO substrates (4 × 1 cm^2), were partly dipped (3 cm) vertically inside the solution. The system was then rubber stoppered. A solution of thiourea (2.5 mL, 0.6 M) was added to the solution with a syringe. The final pH value of the solution was ~ 10.3. The deposition process was continued for different times 30, 45 and 60 min. The 60 min time showed electrodes with preferred characteristics and was therefore used unless otherwise stated.

The CdS films were then taken, washed with distilled water and dried by washing with ethanol and stored in a dry box. Annealing was performed in a horizontal tube furnace under nitrogen for 60 min at 250 °C. This temperature was chosen based on earlier studies as being the suitable annealing temperature for CdS films [25]. The annealed films were slowly cooled back to room temperature with a ramp rate of −1.5 °C/min. Film thickness was calculated gravimetrically by calculating amount of CdS deposited onto each electrode. Thicknesses were 400–600 nm for the freshly prepared film, 300–500 nm for the recycled film with stirring and 200–500 nm for the recycled film with no stirring.

For comparison purposes, fresh CdS electrodes were prepared by CBD using authentic starting materials using literature methods as described above [19,20]. The electrodes were prepared using stirring during deposition as described earlier and annealed at the desired temperature 250 °C.

2.4. The photo-electrochemical (PEC) experiment

PEC study was performed in a three-electrode cell. A PAR 263A

Potentiostat/Galvanostat was used to study the PEC characteristics. The CdS film electrode was used as the working electrode, and a pre-cleaned platinum sheet was used as the counter electrode. The counter electrode was connected to the internal reference cell of the potentiostat. By calibrating the internal cell with an Ag/AgCl reference as described earlier [26], it resembled the NHE reference electrode. A poly sulfide $NaOH/S^{2-}/S_x^{2-}$ system, involving Na_2S (0.10 M), 0.10 M NaOH (0.1 M)/S (0.10 M), was used as a redox couple [19,27]. The Na_2S used was recovered from the waste CdS samples as described above. To avoid oxygen, high purity nitrogen (99.9999%) was bubbled through the solution for 5 min, and was then kept bubbling above the solution throughout each measurement [19]. The illumination intensity at the electrode was 38,000 lx (equivalent to 0.0056 W cm^{-2}).

Photocurrent vs. potential (*I-V*) plots were measured using the CV mode. The measured photo *I-V* plots were divided by exposed CdS electrode surface area to obtain photocurrent density vs. potential (*J-V*) plots. Other PEC characteristics were extracted from the *J-V* plots.

Stability of different CdS electrodes was studied by exposing the electrode to low intensity incident light (0.001 W/cm^2) so as to avoid overheating of the PEC cell. Illumination was continued for 180 h using 0.0 V applied bias. The value of the short circuit current density (J_{SC}) was monitored with time.

3. Results and discussion

Cd^{2+} ions, S^{2-} ions and glass/FTO substrates can all be recovered from waste CdS electrodes. The recovered Cd^{2+} ions were used to prepare new recycled CdS electrodes by the simple CBD methods onto the recovered glass/FTO substrates. Atomic Absorption Spectroscopy (AAS) was used to calculate Cd^{2+} ion concentration in recovered solutions.

The recovered Glass/FTO substrates resembled original fresh Glass/FTO with no significant changes in properties. Both fresh and recovered FTO films showed resistivity values of ~7 Ω/sq, in congruence with vendor's specifications. For confirmation purposes, solid state electronic spectra and photoluminescence emission spectra (excitation wavelength 320 nm) were measured for both fresh and recovered Glass/FTO substrates. Figs. S1 and S2 show similarities between the two systems,

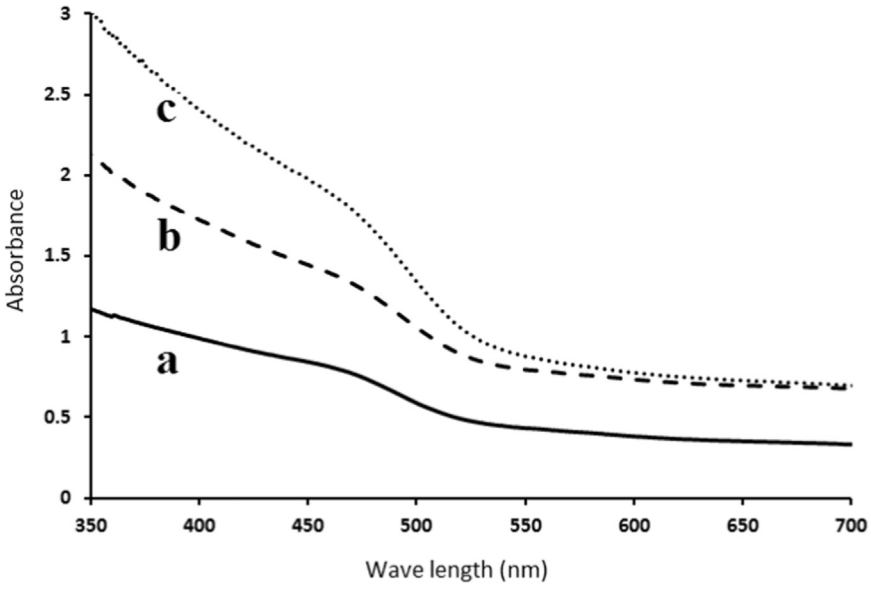

Fig. 1. Solid state electronic absorption spectra measured for different glass/FTO/CdS film electrodes: a) Recycled with stirring, b) Freshly prepared, and c) recycled with no stirring. All measurements were made using glass/FTO for baseline.

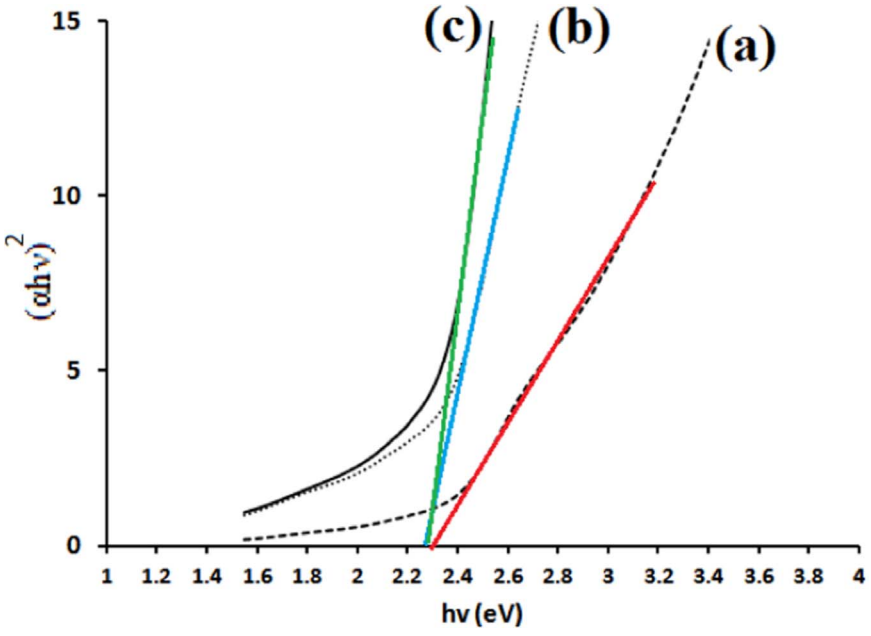

Fig. 2. Tauc plots, assuming direct band gap, for different Glass/FTO/CdS electrodes a) Recycled with stirring, b) Freshly prepared and c) recycled with no stirring.

with no noticeable differences. The resemblance between the two systems is not un-expected, since Glass/FTO substrates are normally pre-cleaned with acidic solutions before any depositing other materials.

After deposition, the remaining Cd^{2+} concentration was calculated by AAS. Different deposition times used showed different amounts of Cd^{2+} ions deposited in each case. Film thickness was studied gravimetrically using the values of Cd^{2+} ion concentrations deposited onto the glass/FTO, assuming CdS density to 4.82 g/cm^3. The film thickness varies with deposition time. The 60 min deposition time yields films with higher thickness. For confirmation purposes, preparations were repeated two or three times.

The results below show how the recycled CdS electrodes resemble the freshly prepared counterparts in many ways. The results also show that the CdS electrode characteristics may also be affected by stirring the preparation solution during CBD process as described below.

3.1. CdS film characterization

Solid state electronic absorption spectra were measured for both fresh and recycled CdS films, with and without stirring, as shown in Fig. 1. The spectra showed an absorption edge at about 530 nm for the recycled and fresh films. The Tauc plots were constructed to measure the value for the band gap. The $(\alpha h\nu)^{2/n}$ was plotted against $(h\nu)$, where α is absorptivity, h is Planck's constant, ν is frequency and n = ½ for direct allowed bands, Fig. 2. The electrodes showed an approximate band gap value 2.3 eV for the fresh and the recycled electrodes,

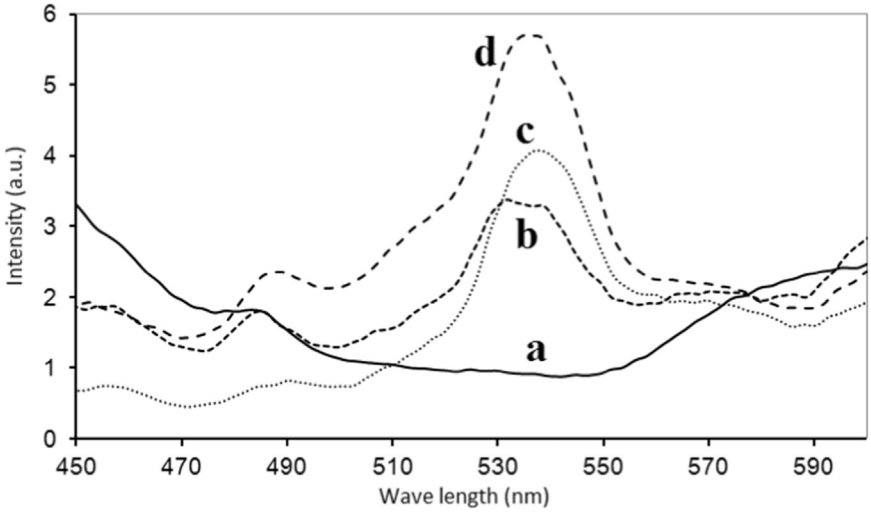

Fig. 3. PL emission spectra measured for different Glass/FTO/CdS films: a) glass/FTO, b) Recycled with stirring, c) Fresh and d) Recycled with no stirring.

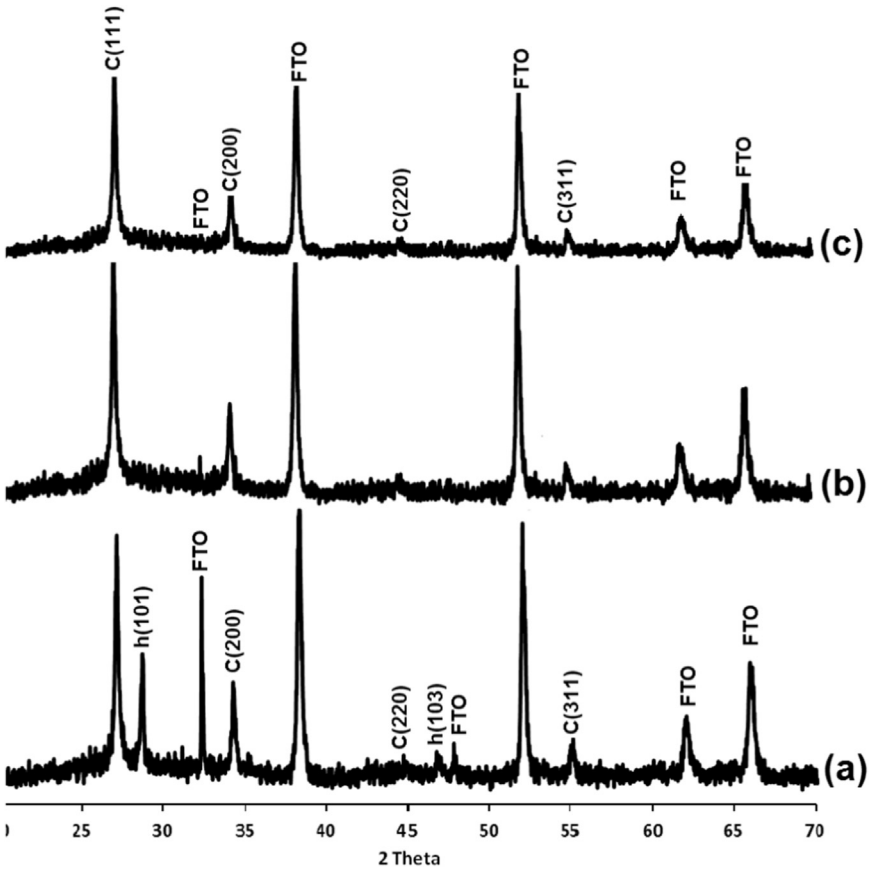

Fig. 4. XRD patterns measured for different Glass/FTO/CdS electrodes: a) Recycled with stirring, b) Recycled with no stirring and c) Freshly prepared.

assuming a direct band gap. All band edge values are within the range reported for CdS polycrystalline films [28–30]. The spectra confirm resemblance of the recycled electrodes with freshly prepared ones.

Photoluminescence (PL) spectra were measured for fresh and recycled CdS electrodes, as shown in Fig. 3. The spectra show emission bands at ~ 535 nm for the electrodes. This is equivalent to a band gap value of 2.3 eV. The value is consistent with that calculated from electronic absorption spectra shown above. The value is also consistent with literature values [31,32]. Other emissions at longer wavelengths are attributed to overtones and crystal deficiencies.

XRD patterns were measured for the fresh and recycled electrodes, as shown in Fig. 4. The presence of FTO (with known reflections at 2ɵ = 32.29°, 38.19°, 47.74°, 51.81°, 62.02° and 65.98°) is observed. The occurrence of the FTO reflections here is attributed to the thin nature of the CdS film onto FTO film. The figure shows that all CdS films exhibit nano size particles, since all films exhibited relatively low height and wide reflections. Calculations based on the Scherrer equation show that the particle sizes are approximately in the range 20–25 nm for different films. Based on literature values, the films dominantly involve cubic phase. This is evident from the positions for the cubic cell planes (111) at 2ɵ = 27.00, (200) 34.24, (220) 44.57, and (311) at 54.73°. The XRD patterns thus confirm that the recycled films, like the fresh one, involve CdS poly-crystals. A closer look at Fig. 4 shows that the reflection heights for the film recycled with no stirring resemble those for the recycled one with stirring. Moreover, the figure shows that with stirring, the reflections at 28.71° and 46.76° for the planes (101) and (103), respectively, in the recycled film, indicate the existence of the hexagonal cell as an additional phase. Therefore, the XRD results show the advantage of using the non-stirring method in order to prepare films with higher phase purity. All XRD patterns are analyzed by comparison with known literature values [23,33–37].

The SEM micrographs were measured for different CdS electrodes, as shown in Fig. 5. The recycled film with stirring involves agglomerates of CdS with sizes ranging 100–400 nm. The agglomerates involve nano-size particles of ~ 30 nm in diameter. The recycled film with no stirring shows similar micrographs with 100–400 nm sized agglomerates having ~ 30 nm nano-particles. The freshly prepared film involves smaller agglomerates of 100–150 nm, with nano-particles of ~ 30 nm in diameter.

The EDX data show a 1:1 atom ratio for Cd to S, with no remaining Cl or other impurities. The elements F, Sn, Si and O are also available as major components due to the glass/FTO substrates.

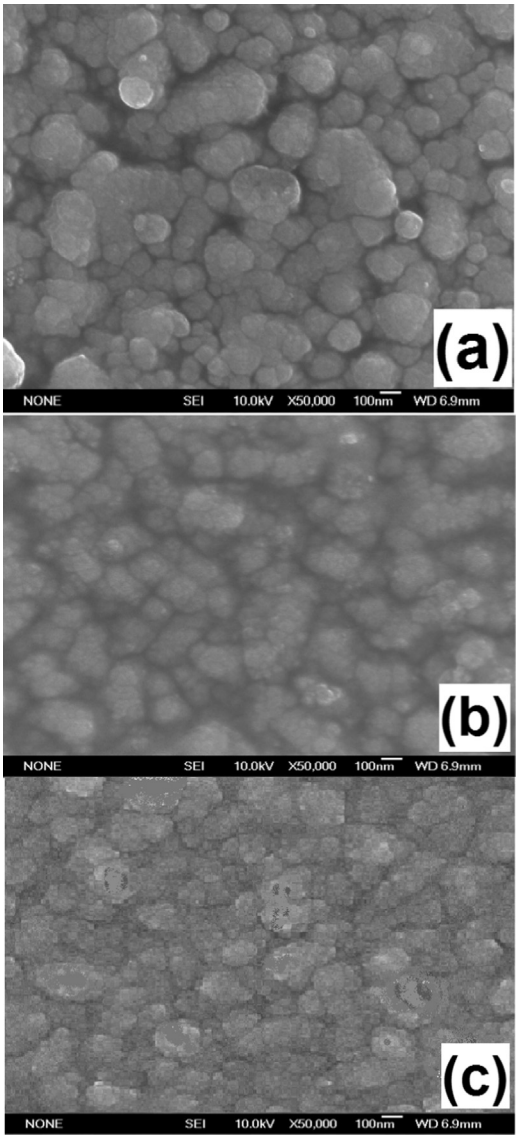

Fig. 5. SEM micrographs measured for different Glass/FTO/CdS film electrodes: a) Recycled with stirring, b) Recycled with no stirring and c) Freshly prepared.

3.2. CdS film electrode PEC characteristics

The photo-current density vs. potential (*J-V*) plots were measured for different glass/FTO/CdS electrodes. For confirmation purposes, PEC measurements were repeated for same film. Fig. 6 shows photo *J-V* plots for the fresh CdS and recycled CdS electrodes (with and without stirring). The *J-V* plots show that the CdS films exhibit n-type semi-conductivity, in congruence with literature [29,38,39]. The PEC characteristics were extracted from the figure and summarized in Table 1. The Table shows values of short circuit current density (J_{SC}), open circuit potential (V_{OC}), fill factor (*FF*) and the conversion efficiency (η%).

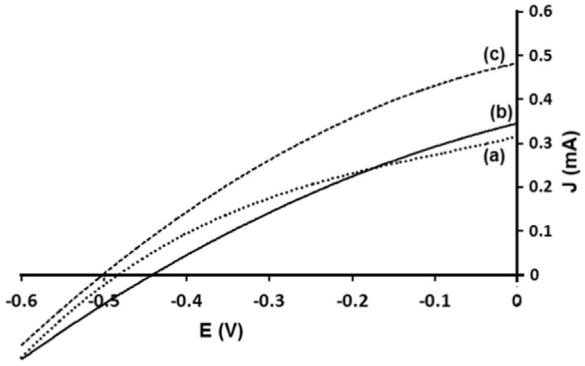

Fig. 6. Photo J-V plots for different Glass/FTO/CdS electrodes a) Recycled with stirring b) Freshly prepared c) Recycled with no stirring.

Entries (1) and (3) of Table 1 show that the recycled CdS electrode with stirring has comparable PEC characteristics with the freshly prepared electrode, in terms of V_{OC}, J_{SC}, *FF* and η%, with slightly lower values. Just like many other metal chalcogenide film electrodes reported in literature [19,20,37,40,41], both electrodes show low PEC characteristics. This should not be counted against the recycled electrode in this work; having in mind the fresh CdS counterpart itself exhibits low conversion efficiency. This is why metal chalcogenide film electrodes are subject to heavy study aiming at enhancing their PEC performance [25,26,42–44]. Similarly the recycled electrode should also be considered in future enhancement activities. A simple method has been followed for this purpose here. Table 1 shows that changing the deposition conditions for the recycled electrode helps achieving this goal. Deposition with no stirring yields recycled CdS electrodes with

Table 1
PEC characteristics for different Glass/FTO/CdS electrodes.

Entry number	Electrode description	V_{oc} (V)	J_{SC} (A/cm^2)	FF % (± 10%)	η% (± 10%)
1	Recycled with stirring	−0.474	0.314×10^{-3}	17.05255	0.7554
2	Recycled with no stirring	−0.500	0.500×10^{-3}	33.7240	1.5940
3	Fresh	− 0.45	0.350×10^{-3}	20%	0.80

*All values were measured at room temperature under nitrogen atmosphere, using polysulfide redox couple. $\eta\% = [(\text{maximum observed power density})/ (\text{reach-in power density})] \times 100\%$. $FF = [(\text{maximum observed power density})/ J_{sc} \times V_{oc}] \times 100\%$. All electrodes were annealed at 250 °C.

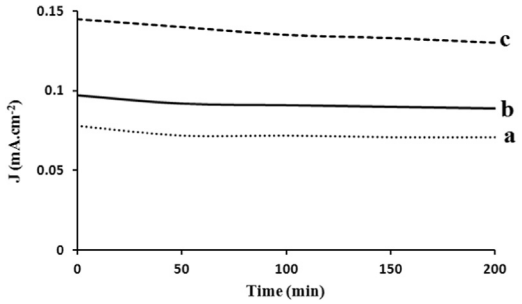

Fig. 7. Plots of J_{SC} value with time showing stability of different Glass/FTO/CdS electrodes under PEC conditions: a) Recycled with stirring, b) Fresh and c) Recycled with no stirring.

higher PEC characteristics, as shown in entry (2) of the Table. Conversion efficiency of ~ 1.6% is exhibited by this electrode. This resembles or even exceeds the relatively low values known for other CBD prepared CdS electrodes [19,20,37,40,41,44]. Efficiency of less than 7.5% was reported for Bi_2MoO_6/CdS composite electrodes for light wavelength of 450 nm [45]. At longer wavelengths (550 nm), the efficiency considerably decreased to less than 2%. Thus, metal chalcogenide electrodes normally exhibit low conversion efficiency, and the recycled CdS electrode is no exception.

The effect of stirring and no stirring on polycrystalline electrodes prepared by CBD has been reported for CdS [46,47] and other systems [48]. Stirring is expected to yield more uniform particles and better contact between the CdS and the FTO layers [47] while in other reports [49] the non-stirred deposition may yield films with preferred characteristics. Stirring is expected to speed up the deposition. Under the working conditions here, electrodes deposited with no stirring showed better characteristics. As observed from XRD patterns, Fig. 4, the electrode prepared with stirring involves mixed cubic and hexagonal phases. This could be the reason for the observed efficiency lowering in the electrode prepared with stirring compared to the one with no stirring. Any shortcomings in poor contact or surface uniformity that may occur in case of no-stirring can be cured by annealing at 250 °C [50,51].

The stability of different CdS electrodes was studied. The electrodes were kept under constant illumination intensity (0.001 W/cm^2) and zero applied bias for more than 3 h. The value of J_{SC} was measured with time, and J_{SC} vs time plots were constructed as shown in Fig. 7. The Figure shows that the recycled electrode with stirring exhibits slightly lower J_{SC} value than the freshly prepared one. The electrode prepared with no stirring exhibits higher J_{SC} value. As per stability with time, all electrodes show lowering in J_{SC} with time. This indicates that the CdS electrodes are relatively unstable under PEC conditions. Photocorrosion of the CdS electrode is expected by accumulation of holes, in the conduction band, upon excitation of electrons. Earlier study showed similar behavior by CdS electrodes [19,20,44]. Such behavior is expected for CdS and other metal chalcogenides, since semiconductors with narrow band gaps exhibit lower stability than those having wider band gaps [52]. Therefore, recent strategies suggested to enhance efficiency and stability of metal chalcogenide electrodes such as CuS [43], CuSe [26], CdS [19,20,25] and CdSe [42,44], are worth to consider for recycled CdS film electrodes.

Collectively, the results show the feasibility of recovering Cd^{2+} ions, S^{2-} ions and glass/FTO substrates from waste CBD electrodes. The recovered Cd^{2+} ions can be used to deposit new electrodes that can achieve comparable PEC characteristics with freshly prepared ones. Further enhancement is possible here simply by using no stirring. Other future enhancement can also be achieved by following simple methods. Work is underway here to obtain recycled CdS electrodes by simple methods with higher conversion efficiencies. Controlling rate of cooling the annealed electrode, coating with electro-active matrices and controlling film thickness are being investigated.

4. Conclusion

Waste CdS film electrodes can be used to recover Cd^{2+} ions, S^{2-} ions and glass/FTO substrates. The recovered materials can be used to produce recycled CdS electrodes with characteristics and PEC properties that are comparable to freshly prepared ones. Further enhancement in characteristics can be achieved by simple techniques such as stirring the deposition bath. Using other simple strategies to enhance the recycled electrodes is worth to consider.

Acknowledgements

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at http://dx.doi.org/10.1016/j.mssp.2017.10.045.

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