

# Release of metal pollutants from corroded and degraded thin-film solar panels extracted by acids and buried in soils

L.C. Su<sup>a</sup>, H.D. Ruan<sup>a,\*</sup>, D.J. Ballantine<sup>a</sup>, C.H. Lee<sup>a</sup>, Z.W. Cai<sup>b</sup>

<sup>a</sup> Environmental Science Program, Division of Science and Technology, Beijing Normal University-Hong Kong Baptist University United International College, Zhuhai, China

<sup>b</sup> Department of Chemistry, Faculty of Science, Hong Kong Baptist University, Kowloon, Hong Kong



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## ABSTRACT

Thin-film solar panels (TFSPs) are widely used in integrated photovoltaic and solar power systems because of their perfect photovoltaic characteristics and ductility. These panels differ from the traditional silicon-based solar panels, in that the metal thin-film layers contain some potentially toxic metals such as zinc (Zn), copper (Cu), nickel (Ni), gallium (Ga), lead (Pb), indium (In) and chromium (Cr). In this study, we examined the environmental pollution that might be caused by disposing of TFSP as domestic trash at the end of their useful life. We used acid extract to simulate metal leaching toxicity and acidic corrosion, and then buried TFSPs in three types of soils to determine if metals might be released into the soil. Our results indicated that the amounts of dissolved metals increased as both the contact time with the acid and the acid concentration in the solution increased during nitric acid extraction. Heavy metals were released from TFSPs in the burial experiment, and the rates of metal release changed with variations in both the amounts of TFSPs in the soil and the soil properties. The increased concentrations of heavy metals such as Zn, Cu, Ni, Ga, Pb, In and Cr in soil samples were correlated to the amounts of TFSPs added. The results of this study confirmed that, when buried, TFSPs polluted the soil.

## 1. Introduction

Compared to traditional fossil fuels, solar power is considered a clean and renewable energy source with zero carbon emissions (Rahim et al., 2012; Can Sener et al., 2018). In 2015, 234 GW (GW) of photovoltaic (PV) capacity were installed globally (Mondal and Denich, 2010; Kumar Sahu, 2015; Urban et al., 2016). In China, the total energy yield from solar cells grew rapidly from 5 GW in 2007 to 30 GW in 2012, and then to 43 GW in 2015 (Urban et al., 2016). In the USA, solar energy generation grew by about 22% in 2010, with new PV modules generating a total of 8 GW installed in 2015, and continues to grow (Zhao et al., 2013; Urban et al., 2016; Archer et al., 2017). It is pertinent that the market share of thin-film solar panels (TFSP) is increasing more rapidly than that of traditional silicon PV panels because of their perfect PV characteristics and ductility. Thin-film solar panels comprised of single-crystalline GaAs cells (28.8%), polycrystalline CdTe thin-film cells (21.5%), perovskite cells (21.0%), and Cu (In,Ga)Se<sub>2</sub> (known as CIGS) cells (21.7%) offering excellent light absorption and high efficiency (Polman et al., 2016). Lee and Ebong (2017) reported that the module efficiencies of CIGS and CdTe TFSPs almost rival that of crystalline solar cells, which currently account for more than 55% of

the market share of TFSPs. The structure (cross section) of a CIGS TFSP is illustrated in Fig. 1. A typical CIGS TFSP contains various layers, including a glass cover, Mo layer, p-type Cu (In,Ga)Se<sub>2</sub> layer, n-type CdS/ZnS layer, TCO(HR-ZnO/n + -ZnO) layer, and an ethylene vinyl acetate (EVA) film, which is always a brown color with a metallic luster. Crystalline silicon or thin-film PV modules have a lifetime between 20 and 30 years and, despite their obvious benefits, their disposal at the end of their useful life may be problematic (Raugei et al., 2007; Shukla et al., 2017). Quite clearly, this is an increasingly urgent issue as there may be as much as 9.57 million tons of end-of-life PV panels to be disposed of by 2050, and the rapid spread of solar power systems means that the potential waste problem is set to increase sharply in the future (Monier and Hestin, 2011).

The vast quantity of waste from spent systems will need to be dealt with properly to avoid environmental problems, and while various solutions for recycling or recovering material from PV cells have already been proposed, researchers are still searching for suitable environmentally friendly and economical methods for recycling TFSPs (Perez-Gallardo et al., 2017). Because of the complexity associated with recycling, TFSPs are generally treated as construction trash, with burial as the preferred disposal option. It is acknowledged that TFSPs should

\* Corresponding author.

E-mail address: [hruan@uic.edu.hk](mailto:hruan@uic.edu.hk) (H.D. Ruan).

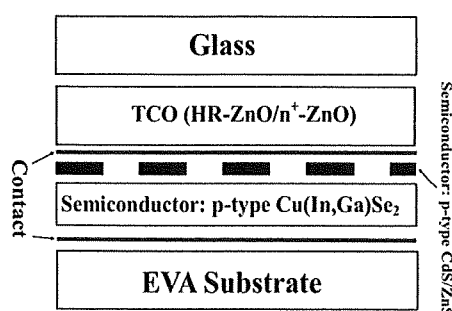


Fig. 1. Structure of a typical CIGS TFSP (cross section); TCO stands transparent conductive oxide, and EVA stands ethylene vinyl acetate.

not be disposed of along with domestic trash as the internal thin-film paint coat contains hazardous metals. Unlike other electrical products, TFSPs are built to resist harsh natural environments, as the toxins in their electrical components are continuously exposed to the environment. The metal thin-film paint coat on a TFSP is well protected by the glass cover and EVA (Fig. 1), but it may become a source of pollution once the glass cover or EVA film is broken. If such damage occurs during the disassembly and transfer stages in a solid waste treatment stream, the internal thin-film paint coat, comprised of the Al/Ni grid, Mo, p-type Cu(In,Ga)Se<sub>2</sub>, n-type CdS/ZnS and TCO(HR-ZnO/n<sup>+</sup>-ZnO), may be exposed to the environment. Oxygen and water may then permeate into the damaged areas and stimulate corrosion of the thin-film paint coat, resulting in the release of metals.

Previous studies have shown that acid solutions can simulate the leaching toxicity of metal release under acidic environmental conditions, and accelerate the release of metal contaminants from solid waste (Arain et al., 2008; Kazi et al., 2009; Feng et al., 2015). Deng et al. (2009) reported that the expulsion efficiencies of copper (Cu), zinc (Zn), and lead (Pb) from sludge grew as nitric acid concentrations increased (0–0.65 M) and the ideal nitric acid concentration for extracting these metals was 0.325 M (Tyagi et al., 2014). It has also been reported that the release of trace metals from burial of e-waste could contaminate the soil, and the acidic process (vitriolic acid, pH = 1) is the best way to remove cadmium (Cd), chromium (Cr), nickel (Ni) and Zn from domestic and industrial solid waste (Lo and Chen, 1990; Cui et al., 2017). The disposal of TFSPs appears rather harmless and straightforward as long as the toxins inside remain protected by the toughened glass and EVA film. However once the protective layers of TFSPs are damaged, as is inevitable during the waste treatment process, there is the potential for release of heavy metals and other toxins from TFSPs to the surrounding environment, with consequences for soil and water quality. Other types of wastes have been reported to cause similar environmental pollution. For example, investigations of an electronic waste dumpsite showed that the total mean concentrations of heavy metals in the soil decreased with depth in the soil profile and with distance from the dumpsite (Khan et al., 2008; Olafisoye et al., 2013; Adamcová et al., 2017). There are also concerns that the hazardous and toxic metals in the PV modules, such as Cu, Zn, Pb, Cd and selenium (Se), may have serious impacts on human health (da Silva et al., 2017a; da Silva et al., 2017b; Khidkhan et al., 2017; Ngole-Jeme and Fantke, 2017). To date, there have been few studies of the environmental pollution that arises as TFSP is broken down or degraded. One study reported that the predicted environmental concentrations (PEC) of Cd (173.4 µg L<sup>-1</sup>), molybdenum (Mo) (9.9 mg/L), and Se (9.4 µg L<sup>-1</sup>) released from thin-film (CIGS) panels during acid rain runoff experiments were considerably greater than the acute toxicity concentrations for some aquatic animals (Zimmermann et al., 2013). Also, very few studies have reported the mechanisms that control the release of toxins into the soil when TFSPs are disposed of. Because of its potential as one of the most promising semi-conductive materials in the thin-film industry, the toxic effects of CIGS and other similar semi-conductive

materials need to be investigated. The aim of this study therefore was to determine the mechanisms that controlled metal release from CIGS TFSP material in acidic solutions, and the potential for pollution when TFSP is buried in different types of soils.

## 2. Materials and methods

### 2.1. Thin-film solar panel extraction

#### 2.1.1. Preparation of thin-film solar panel material

The TFSPs were obtained from a solar energy company in Zhuhai, China. We removed the metal frames, electric cables, and plastic debris (Fig. 1) and then crushed the panel material into small pieces (approximately 9.5 mm × 9.5 mm) with a hammer wrapped in plastic film. The crushed materials were rinsed with deionized (DI) water and air-dried at room temperature for 48 h.

#### 2.1.2. Determination of metal constituent in thin-film solar panel material

The metals in TFSP semiconductor layers were extracted by microwave-assisted digestion (Microwave digester MDS-10, Sineo, Shanghai). Approximately 1.00 g of homogenized TFSP semiconductor material that had been ground to less than 0.15 mm was mixed with 10 mL strong acid (4:1 concentrated 65% HNO<sub>3</sub> and 30% H<sub>2</sub>O<sub>2</sub> (v/v)) in an airtight Teflon tank (effective volume 50 mL) and heated to 180 °C in the microwave digester (Sineo, MDS-10, Shanghai, China) for 20 min. After digestion the supernatant was separated from the solid particles with a 0.22 µm filter and diluted to 50 mL (Krishnamurthy et al., 1976; Farkas, 1993). The concentrations of metals (Al, Ni, Zn, Cu, Na, Ga, V, In, Fe, Pb, Rb, Se, Mn, Mo, Sr and Cr) in semiconductor layers were determined using ICP-OES (Perkin-Elmer Optima 2100 DV, USA).

#### 2.1.3. Solid waste leaching toxicity

The leaching toxicity was determined followed the HJ/T299-2007 Solid waste-Extraction procedure for leaching toxicity-Sulphuric acid & nitric acid method. In this experiment, approximate 100.00 g of homogenized TFSP materials that had been further ground to approximate 3 mm were mixed well with 2.00 L leaching reagent (two drops of concentrated 2:1 98% H<sub>2</sub>SO<sub>4</sub> and 65% HNO<sub>3</sub> (v/v) in 1 L of DI water, pH ≈ 3.25). The mixture in the flask was agitated on a shaker with a shaking speed of 30 r/min for 18 h at 23 °C, and then filtered with a 0.22 µm filter. The metal concentrations in the solution after filtration and acidification (by HNO<sub>3</sub>, to pH less than 2) were determined using ICP-OES (Perkin Elmer Optima 2100DV).

#### 2.1.4. Nitric acid extraction

We carried out pre-experiment tests to determine which acid, out of sulphuric acid, ortho-phosphoric acid, hydrochloric acid, nitric acid, and ethylic acid at concentrations of 0.7, 1.4, 2.1, 2.8 and 3.5 M, showed the best ability to corrode TFSP materials and found that the corrosion was best with nitric acid solution (see supplementary material). We then simulated a range of acidic conditions at different nitric acid concentrations and studied the influence of time and acidity on the degree of corrosion of the TFSP materials. Specifically, we placed 10.00 g of the crushed TFSP material (less than 9.5 mm × 9.5 mm), from which the Ni/Al grid was removed to permit a better examination of corrosion for thin-film paint coat, into 250 mL conical flasks and extracted these samples for 1, 6, 12, 24, 48, and 72 h with 100 mL nitric acid at 5 different concentration levels (0.7, 1.4, 2.1, 2.8 and 3.5 M) by shaking (150 rad/min) at room temperature (20 °C). The supernatant was then separated from the solid particles with a 0.22 µm filter and collected in polytetrafluoroethylene (PTFE) bottles. The concentrations of metals (Zn, Ni, Al, Cr, Ga, Pb, Cu and In) in the solution were determined using ICP-OES.

**Table 1**  
The properties of the soils used in the experiment.

	Synthetic soil	Mollisol	Oxisol
pH	7.2	5.6	3.9
Organic matter (%)	9.04%	5.30%	8.21%
Sand + silt (> 2 μm)	81.98%	56.24%	49.64%
Clay (< 2 μm)	8.98%	38.46%	42.15%
Pb (mg kg <sup>-1</sup> )	4.00	27.10	193.60
Cu (mg kg <sup>-1</sup> )	12.05	18.80	294.25
Zn (mg kg <sup>-1</sup> )	40.00	28.15	95.50
Ni (mg kg <sup>-1</sup> )	10.80	2.80	20.42
Cr (mg kg <sup>-1</sup> )	7.04	13.45	18.53
Ga (mg kg <sup>-1</sup> )	3.15	9.50	2.05
In (mg kg <sup>-1</sup> )	6.60	1.41	5.28

## 2.2. Soil burial experiment

### 2.2.1. Preparation of soil sample

Two soil samples were collected respectively from the upper 20 cm of soil profiles in Zhuhai and Shaoguan, both in Guangdong Province, China. The sample from Zhuhai was a neutral Mollisol that represented a “unpolluted soil” while the sample from Shaoguan was an acid Oxisol with a low pH and high heavy metal background concentrations that represented a “polluted soil”. We also prepared a synthetic soil, which was a commercial soil with a neutral pH and low heavy metal concentrations that represented a “garden soil”. We air-dried the collected soil samples at room temperature, ground them to less than 2 mm, and then stored in high-density polyethylene (HDPE) buckets. All the soil samples were heated at 121 °C for 24 h to eliminate indigenous microorganisms to avoid bio-leaching of metals. The major properties of these soils are described in Table 1.

### 2.2.2. Thin-film solar panel burial experiment

Evenly mixed soil samples (2.0 kg) were placed in HDPE pots (250 \* 150 \* 200 mm). Each pot had 2 small holes (φ 5 mm) in its base and a drainage pan underneath into which water drained. This drainage water was returned to the original pot once a day. Different amounts of the crushed TFSP material (with Ni/Al grid) were mixed evenly with the soil samples (0, 50, 100, 150 and 200 g were mixed with 2.0 kg of soil) in the pots, with three pots for each treatment. The samples were left in an open cultivation area under natural environmental conditions (25 ± 5 °C) for 60 days and were soaked with DI water once a day to keep the soil moist (60 ± 5%). After burial, a small amount of the soils in which the remaining TFSP materials had been completely taken out was collected from each pot. These soil samples were air dried at room temperature, then ground to 0.15 mm and stored in a refrigerator at 4 °C. Homogenized soil materials (approximately 200 mg) with 10 mL strong acid (4:1 concentrated 65% HNO<sub>3</sub> and 30% H<sub>2</sub>O<sub>2</sub> (v/v)) in an airtight Teflon tank (effective volume 50 mL) were heated at 180 °C in the microwave digester (Sineo, MDS-10, Shanghai, China) for 20 min. After digestion the supernatant was then separated from the solid particles with a 0.22 μm filter and diluted to 50 mL (Krishnamurthy et al., 1976; Farkas, 1993). The concentrations of metals (Zn, Ni, Al, Cr, Ga, Pb, Cu and In) were determined using ICP-OES (Perkin-Elmer Optima 2100 DV, USA). The experiments were performed in triplicate. A certified standard reference material GBW07403(GSS-3), obtained from the National Institute of Metrology (NIM), China, was used in the digestion and analysis as part of the QA/QC protocol. Reagent blanks and analytical triplicates comprised 10% of the total samples were used where appropriate to test the accuracy and precision of the analysis. The recovery rates were around 92 ± 6% for all of the metals in the soil reference material (GBW07403(GSS-3)).

## 2.3. Data analysis

We used the Pollution Index (PI) and Nemerow Contamination

Index (P<sub>N</sub>) to quantify the pollution and to evaluate the metal pollution levels for soil samples.

### 2.3.1. Pollution index

The PI ratio was calculated as outlined in the Technical Specifications for Soil Environmental Monitoring HJ/T 166 (2004), and expressed in Equation (Eq.) 2-1.

$$PI = \frac{[C]_{\text{heavy metal}}}{[C]_{\text{background}}} \quad (2.1)$$

where [C]<sub>heavy metal</sub> is the heavy metal concentration in a soil sample and [C]<sub>background</sub> represents the heavy metal concentration in a blank soil (background).

### 2.3.2. Nemerow Contamination Index

The Nemerow pollution index (P<sub>N</sub>) is exceptionally sensitive and resilient in eclipsing properties, making it a good general-use index on classifying contamination levels of soils (Cai et al., 2015). It considers all the independent pollution indexes and highlights the importance of elements that cause contamination.

$$P_N = \sqrt{\frac{PI_{\text{average}}^2 + PI_{\text{max}}^2}{2}} \quad (2.2)$$

where PI is the pollution index ratio, PI<sub>average</sub> and PI<sub>max</sub> are the average and maximum PI values for each metal, respectively. The relationships between the value of P<sub>N</sub> and level of pollution are shown in Table 2. The P<sub>N</sub> reflects the combined effects of multiple heavy metals in soils, especially the pollutants with the highest PI.

### 2.3.3. Statistical analysis

Statistical analysis was performed with SPSS (Ver. 22). We used analysis of variance (ANOVA) to test differences between the concentrations of different metals at different extraction times during the acid extraction experiment, and the relationships between the metal concentrations and the different leaching times for the various soils. Means were separated using the least significant difference option with α = 0.05. The correlations between addition of crushed TFSP (g) and Pollution Index (P<sub>i</sub>) and Nemerow Contamination Index (P<sub>N</sub>) of soils are analyzed using Pearson rank order correlation analysis.

## 3. Results

### 3.1. Determination of metal constituent in thin-film solar panel material

The metal constituents in the TFSP semiconductor materials are shown in Fig. 2. As the main elements in Ni/Al grids, aluminum (Al) and Ni, with concentrations of 1270 and 650 mg/kg, respectively, were the two most abundant metals. As expected, Zn, Cu, Ga, and In, the important components of a thin-film paint coat (e.g. TCO (ZnO), p-type Cu(In,Ga)Se<sub>2</sub>) used for generating electricity, were also present at high concentrations. The main source of sodium (Na) is glass (Fig. 1), while iron (Fe) and vanadium (V) were present in the residual metal frames that were removed in the pre-treatment. The remaining metals, detected at trace concentrations, are added to TFSPs during manufacturing to optimize the optical and photovoltaic properties, or to

**Table 2**  
Nemerow Contamination Index (P<sub>N</sub>) for evaluating soil pollution.

Level	Nemerow Contamination Index	Pollution Level
I	P <sub>N</sub> ≤ 0.7	Nil
II	0.7 < P <sub>N</sub> ≤ 1.0	Light
III	1.0 < P <sub>N</sub> ≤ 2.0	Mild
IV	2.0 < P <sub>N</sub> ≤ 3.0	Medium
V	P <sub>N</sub> > 3.0	Heavy

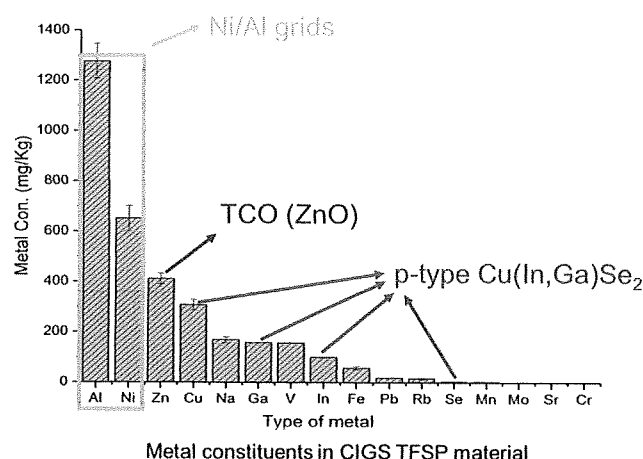


Fig. 2. Metal concentrations of TFSP after microwave-assisted digestion. (Values in the bar chart are expressed as the mean and error bars represent the SD. Three samples were analyzed for each metal. SD = standard deviation).

prevent corrosion of the paint coats.

### 3.2. Metal leaching toxicity from TFSP material

Fig. 3 demonstrates the metal leaching toxicity of TFSPs. As the main elements in a thin-film panel coat (e.g. ZnO), the concentration of Zn (3.66 mg/L) showed the highest value among all metals. The other important components in Ni/Al grids, aluminum (Al) and nickel (Ni), also showed high concentration of 1.69 and 1.25 mg/L, respectively. Since none of the metal leaching toxicity of TFSP exceeded the related national standard (GB 5085.3–2007 Identification standards for hazardous wastes – Identification for extraction toxicity), the TFSP materials can be defined as solid waste s below the national level of leaching toxicity based on this experiment.

### 3.3. Nitric acid extraction of metals from TFSP material

Fig. 4 shows the four major metal components of TFSP, especially of the thin-film paint coat (without glass cover and Ni/Al grid) after acid extraction. During the first 24 h of extraction, the concentration of Zn in solution increased significantly ( $p < 0.05$ ), after which the

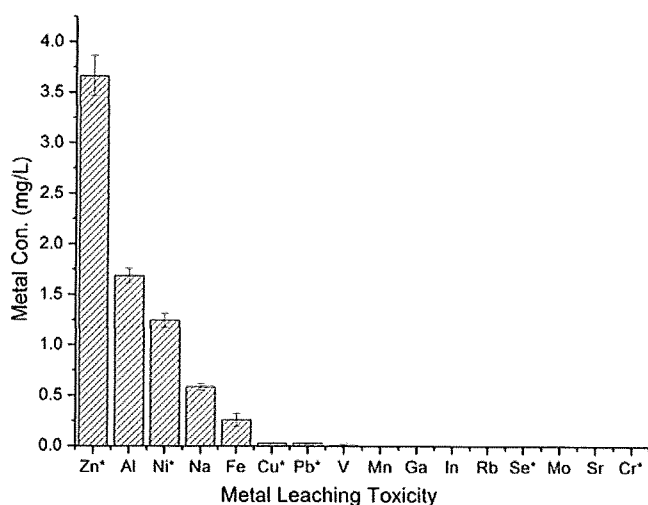


Fig. 3. Metal leaching toxicity of TFSPs. \*Denotes the metals that ought to be targeted during leaching toxicity evaluation. Values of metal concentrations are expressed as the mean with error bars representing the standard deviation (SD). Five replicates were analyzed.

concentrations increased very slowly. As shown in Fig. 4a, the concentration of Zn extracted by 3.5 M nitric acid solution was 30% higher than that extracted by 0.7 M nitric acid during the first hour of extraction. The pattern of Zn extraction was different from those of Ga, In and Cu, as shown in Fig. 4. Zinc showed a rapid increasing trend in the early stages of extraction while Ga, In and Cu followed a contrasting pattern. After extraction for 6 h, Ga, In and Cu were dissolved slowly in the solution, and the increases in their concentrations were not significant ( $p > 0.05$ ). Dissolution of Ga, In and Cu started to increase in 1.4, 2.1, 2.8, and 3.5 M nitric acid solutions between 6 and 24 h of extraction. It was noticed that even Ga and In started to increase in 0.7 M nitric acid solution after 24 h of extraction. After 48 h of extraction, the concentrations of Ga, In and Cu had increased significantly ( $p < 0.05$ ) in all nitric acid concentrations (Fig. 4b, c & 4d), and the dissolution rates of Ga, In and Cu showed obvious increases after 48 h of extraction in 2.1, 2.8, and 3.5 M nitric acid solutions. As shown in Fig. 4d, the Cu concentration peaked after 72 h of extraction, but the Cu peak concentration was much lower than those of other metals. Only 0.64 mg/L of Cu was extracted in 2.1 M nitric acid solution, and after extracting with 2.1, 2.8, and 3.5 M nitric acid solutions, the Cu concentrations were almost the same. The 1.4 and 0.7 M nitric acid solutions showed weaker extraction abilities, and they extracted only half as much Cu as the 2.1 M nitric acid after 72 h.

### 3.4. Thin-film solar panel burial experiment

The metal concentrations in the synthetic soil, Mollisol, and Oxisol increased as the amounts of TFSP added increased over a 60-day burial period. The metals presented the highest concentrations in these three soils were Ga, Zn, Pb, Cu, Ni, In and Cr (Fig. 5).

#### 3.4.1. Burial experiment of synthetic soil

Of the heavy metals studied, the Ni concentration was the highest in the synthetic soil after 60-day burial (Fig. 5a). The concentration of Ni increased 12 times from a background level of 10.8–137 mg/kg in the synthetic soil where 200 g of TFSP (with Al/Ni grid) was added, which indicated that the release of Ni was significantly ( $p < 0.05$ ) correlated to the amount of TFSP added. The background concentrations of Zn and Cu in the synthetic soil were 40 and 13 mg/kg, respectively. For example, after 60 days of burial with addition of 200 g of TFSP, the concentration of Zn in the soil increased to approximately 82 mg/kg. This was approximately twice the concentration in the blank soil sample, and corresponded well with the amount of TFSP added. The Cu concentration was 39 mg/kg in the soil where 200 g of TFSP was added, which was 3 times the concentration in the blank (Fig. 5a). The In and Ga concentrations also increased as the amount of TFSP added increased, from a background concentration of 6.6–16.5 mg/kg for In and 3.2–5.2 mg/kg for Ga in the soil where 200 g of TFSP was added after 60 days of burial (Fig. 5a).

#### 3.4.2. Burial experiment of Mollisol

After burial for 60 days, we found that the concentrations of Zn, Cu, Ni, Ga, In and Cr in Mollisol were higher than those at the beginning of burial period, reflecting the release of metals from TFSP (Fig. 5b). Of these metals, the concentration of Zn was the highest, and had increased from a background level of 28 mg/kg to a maximum of 131 mg/kg as the amounts of TFSP added increased to 200 g. The concentration of Ni was initially 2.8 mg/kg but increased to 13.6 mg/kg, 4 times its background concentration, and the concentration of Pb increased by approximately 18.5% from 27.1 to 32.7 mg/kg in the soil where 200 g of TFSP was added after the 60-day burial, due to soldering that contains Pb was used in TFSP and contributed to Pb release into the soil. The concentrations of Ga and Cr in Mollisol changed only slightly when different quantities of TFSP were added.

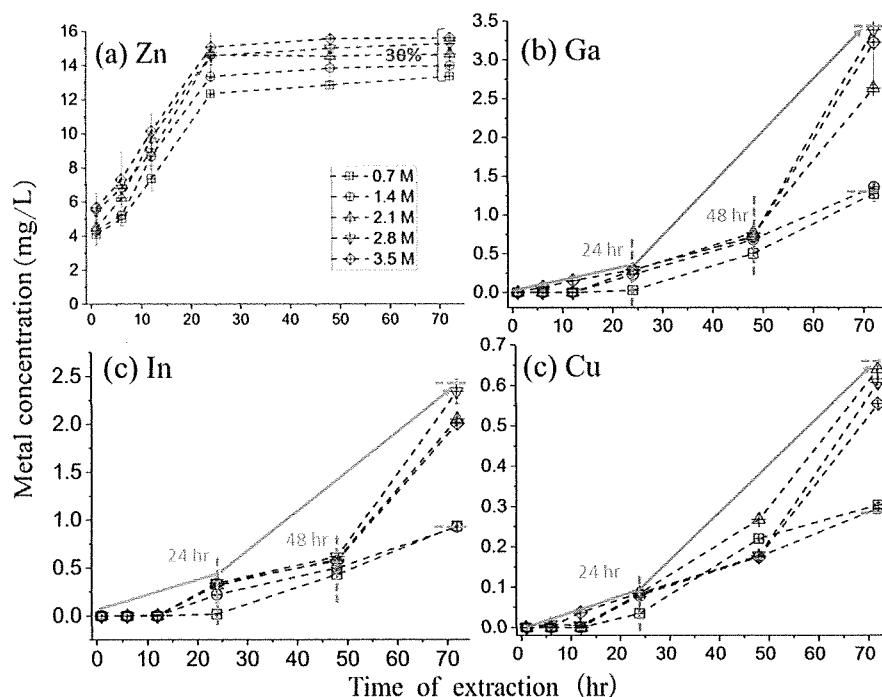


Fig. 4. Metal concentrations vs. extraction time. Legends in (a) indicate the concentrations of nitric acid used for the extraction. Values are expressed as the means with error bars representing the standard deviation (SD). Five replicates were analyzed for each sample.

#### 3.4.3. Burial experiment of oxisol

After burial for 60 days, the metallic luster of TFSP had faded more in Oxisol than in Mollisol or synthetic soil. This suggested that more serious corrosion occurred on TFSP when buried in Oxisol. The main pollutants released were Cu and Zn, which were the main elements in the thin-film paint coat. The concentrations of Cu and Zn increased as the amount of TFSP added to the soil increased from 50 to 200 g (Fig. 5c). The background concentrations of Cu in Oxisol were much higher than those in synthetic soil and Mollisol and increased 29% to 379.79 mg/kg after adding 200 g of TFSP and buried for 60 days. The concentration of Pb was also high, but it was already present in Oxisol at quite a high concentration. The background Pb level, rather than the added TFSP, was perhaps the main contributor to the high Pb concentration in Oxisol.

## 4. Discussion

#### 4.1. Metal leaching toxicity from TFSP material

Although various metals can be extracted by the low concentration of leaching reagent ( $\text{pH} = 3.25$ ) during an 18 h extraction, but the concentrations of these metals did not exceed those as recommended by the national standard. Compared to the metal constituents of TFSP semiconductor materials, very tiny amounts of metals were extracted during such a treatment even if the metallic luster on TFSP materials can be observed clearly. The protective frame (e.g. EVA) resists the thin-film paint coat from acid corrosion, which is the main function it performs under the normally environmental conditions. Analyzing the leaching toxicity of TFSP materials using weak acid solution aims at simulating the pollution arisen by acidic precipitations (e.g. acid rain) when the protective frame is broken after disposal. We considered that in a real natural environment, it has high possibility for the solid waste exposed to such an acidic environment for a long period of time. It is important to investigate the corrosion process of semiconductor materials in TSFPs, which can assist to understand the release of metals after disposal. For this purpose, we designed a series of experiments by using

strong acid extractions to deal with the corrosion of thin-film paint coat.

#### 4.2. Nitric acid extraction of metals from TFSP

The concentrations of Zn extracted with various concentrations of nitric acid over a 72-h period were about 3 times higher than those extracted during the first hour, which indicated that the amount of dissolved Zn increased as both the contact time with acid and the acid concentration in the solution increased. The increase in Zn concentration during the first hour of extraction was attributed to the dissolution of the ZnO paint coat that was used to modify the photovoltaic ability in TFSP. As a novel material in a II-VI semiconductor, TCO (HR-ZnO/n + - ZnO) is extensively used in surface acoustic wave layers (Ie Reinders et al., 2017). Compared to the traditional ITO ( $\text{In}_2\text{O}_3:\text{Sn}$ ) layers, the new series of TCO layers, especially highly conductive undoped ZnO (b-ZnO), offer better conductivity and greater stability at a lower price (Hala et al., 2017). The maximum Zn concentrations after 72 h of extraction were correlated with the different rates of Zn dissolution. The increases in extraction rate between 1 and 24 h was related to the decomposition of ZnO paint coat, while the decreases in extraction rate between 24 and 72 h corresponded with Zn released from the internal components of TFSP.

The dissolution rates of Ga, In and Cu increased slowly between 1 and 24 h of extraction but rapidly from 24 to 72 h, which indicated that these metals were mainly released from the internal parts (the p-type  $\text{Cu(In,Ga)Se}_2$  layer of TFSP as shown in Fig. 1). When the thin-film paint coat is well-protected with the glass and EVA film, the transparent conductive oxide (TCO) film (e.g. ZnO paint coat) that covers the TFSP may also protect against acid corrosion. The amount of Ni extracted during this experiment was tiny, and did not correspond well to the high Ni content in TFSP after microwave-assisted digestion. To study the mechanism of TFSP thin-film paint coat corrosion, the Ni/Al grid on the TFSP used in this experiment was manually removed. The results reflected the fact that high Ni content in TFSP was mainly from the Ni/Al grids.

Our tests at different acidic concentrations and extractions over

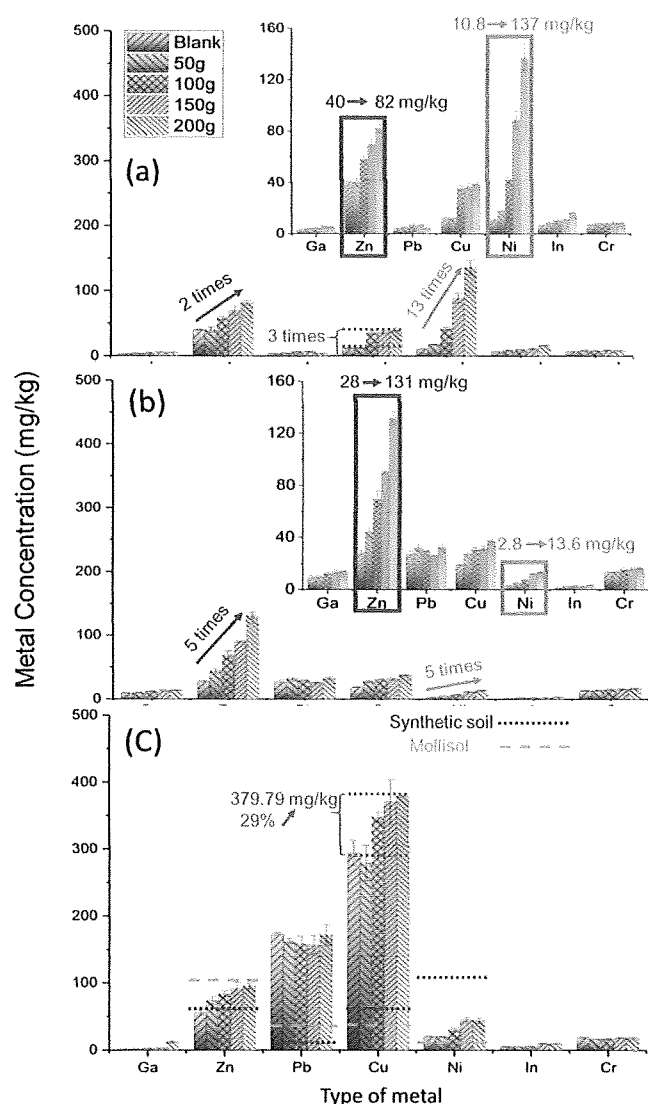


Fig. 5. Metal concentrations in soils contaminated by TFSP: (a) synthetic soil, (b) mollisol, and (c) oxisol. The inset graphs in Fig. 5-a & b have been included to show the results more clearly. Labels in (a) indicate the amounts of TFSP added. Values are expressed as the mean  $\pm$  SD. Samples were analyzed in triplicate.

different periods of time indicated that the metals in TFSP could be released under acidic conditions, and that the duration of the exposure to the acidic environment was an important parameter of influence on the amounts of metals released. Hazardous metals may be released to the environment from the thin-film paint coat inside TFSP when the protective layers are broken, and the release may be accelerated by the acidity of the surrounding environment.

#### 4.3. Thin-film solar panel burial experiment

##### 4.3.1. Burial experiment of synthetic soil

Indium and Ga are key elements in the p-type Cu (In, Ga)  $\text{Se}_2$  layer of a CIGS TFSP, so the increases in their concentrations in the soil indicated that the paint coat of the thin-film was corroded. The internal thin film layer can therefore be corroded easily once the glass or EVA film is broken. The results of increases in the total concentrations of Ga, Zn, Pb, Cu, Ni, In and Cr in the synthetic soil samples as shown in Fig. 5a demonstrate that heavy metals were released from TFSP during

Table 3

Pollution Index (PI) and Nemerow Contamination Index ( $P_N$ ) of soils.

Soil type	Addition of crushed CIGS TFSP (g)				Correlation factor (r)
Synthetic soil	50 g	100 g	150 g	200 g	
$P_{Ga}$	1.26	1.45	1.88	1.64	0.764
$P_{Zn}$	1.01	1.46	1.74	2.05	0.994**
$P_{Pb}$	1.20	1.69	1.72	1.05	-0.159
$P_{Cu}$	1.02	2.96	3.05	3.23	0.837
$P_{Ni}$	1.65	3.92	8.24	12.69	0.990**
$P_{In}$	1.29	1.56	1.70	2.50	0.935*
$P_{Cr}$	1.16	1.14	1.23	1.19	0.593
$P_{AVG}$	1.24	2.17	3.06	3.86	0.999**
$P_N$	1.46	3.17	6.21	9.38	0.992**

Pollution level	Mild	Heavy	Heavy	Heavy	-
Mollisol	50 g	100 g	150 g	200 g	-
$P_{Ga}$	1.06	1.30	1.40	1.47	0.958*
$P_{Zn}$	1.57	2.47	3.22	4.68	0.989**
$P_{Pb}$	1.17	1.10	0.96	1.21	-0.024
$P_{Cu}$	1.47	1.64	1.69	2.01	0.956*
$P_{Ni}$	1.80	2.66	4.30	4.84	0.983**
$P_{In}$	1.86	2.06	1.76	2.55	0.651
$P_{Cr}$	1.35	1.35	1.42	1.63	0.887
$P_{AVG}$	1.49	1.87	2.22	2.79	0.993**
$P_N$	1.69	2.30	3.42	3.95	0.990**

Pollution level	Mild	Medium	Heavy	Heavy	-
Oxisol	50 g	100 g	150 g	200 g	-
$P_{Ga}$	0.94	1.57	1.60	6.04	0.84
$P_{Zn}$	0.92	1.22	1.38	1.51	0.980**
$P_{Pb}$	0.92	0.90	0.89	0.97	0.508
$P_{Cu}$	0.95	1.18	1.26	1.29	0.923*
$P_{Ni}$	0.98	1.50	2.22	2.14	0.929*
$P_{In}$	0.98	1.07	1.86	1.89	0.923*
$P_{Cr}$	0.92	0.92	1.00	0.99	0.861
$P_{AVG}$	0.94	1.19	1.46	2.12	0.968*
$P_N$	0.96	1.39	1.88	4.52	0.901*
Pollution level	Light	Mild	Mild	Heavy	-

<sup>a</sup>  $P_{metal}$  stands for the pollution factor of metal in soil and  $P_{AVG}$  is the average value of  $P_{metal}$ ; and  $P_N$  stands for Nemerow contamination index of the soil. The correlations are between the  $P_N$  and the amounts of TFSP materials added; \*,  $p < 0.05$  and \*\*,  $p < 0.01$ .

the 60-day burial period.

Synthetic soil with addition of 100, 150, and 200 g of TFSP was classified as heavily contaminated ( $P_N$  values of 3.17<sub>100g</sub>, 6.21<sub>150g</sub>, and 9.38<sub>200g</sub>, as shown in Table 3). The Ni concentration in synthetic soil was higher than the limit of the Environmental Quality Standards for Soil in China GB15618 (2018), while the concentrations of other metals did not exceed the standard. Nickel was the primary metal contaminant released from TFSPs when buried in synthetic soil. The different leaching patterns of synthetic soil and Mollisol were mainly attributed to the soil properties, including pH and organic matter. The organic matter in synthetic soil ( $OM_{synthetic} = 9\%$ ) was higher than in Mollisol ( $OM_{mollisol} = 5.3\%$ ), which was added in for enrich the soil nutrient (e.g. peat). The organic matter showed a good Ni adsorption ability, which led to the equilibrium concentration of Ni in synthetic soil much higher than that in Mollisol. It is notice that the concentration of Ni in synthetic soil increased with the amount of TFSP added.

##### 4.3.2. Burial experiment of Mollisol

The background concentration of Zn in Mollisol was approximately 30% less than that in synthetic soil, but after 200 g of TFSP was added and buried for 60 days, the Zn concentration in Mollisol was 1.5 times higher than that in synthetic soil. The concentrations of Ni followed a different pattern. The background concentration of Ni in Mollisol was only 1/3 of the concentration in synthetic soil. After 60 days, the Ni concentration in Mollisol to which 200 g of TFSP was added had

increased from 2.8 to 13.6 mg/kg but was only 10% of the concentration in synthetic soil. After 60 days, the Cu concentration had doubled from 18.8 to 37.7 mg/kg in Mollisol to which 200 g of TFSP was added (Fig. 5b); this was comparable to the concentration of Cu extracted from synthetic soil with 200 g of material added (Fig. 5a). These results suggest that it was easier to extract Zn, but more difficult to extract Ni, from the Mollisols (pH 5.6) than from the synthetic soil (pH 7.2) with TFSP added. According to Barać et al. (2016), soil contamination by heavy metals not only significantly increased the concentrations of heavy metals in the soil but also their mobile and potentially bioavailable amounts (Abdu et al., 2017; Xiao et al., 2017). Our results may reflect the characteristics of these soils, including their pH, humic acid and hydrous ferric oxide contents, particle size and shape, ionic strength, specific surface area, surface bonding, cation exchange capacity, and their affinity to accelerate or retard the release of metals from TFSP. The Mollisol used in this experiment was a kind of natural soil collected in Zhuhai, with less organic matter ( $OM_{\text{Synthetic}} = 9\%$ ;  $OM_{\text{Mollisol}} = 5.3\%$ ) than synthetic soil but with much higher clay content ( $Clay_{\text{Synthetic}} = 8.98\%$ ;  $Clay_{\text{Mollisol}} = 39.46\%$ ). The selective adsorption of Zn on the clay increased the total adsorption capacity of Zn by Mollisol. The Zn concentration was higher than the limit of the Environmental Quality Standards for Soil in China GB15618 (2018) in the soil where 200 g of TFSP was added after 60 days of burial, indicating that the Mollisol was contaminated. Zinc was the primary metal contaminant released from TFSP when buried in Mollisol. According to Strachel et al. (2017), high concentration of Zn in contaminated soil can significantly modify the soil properties by accelerating or retarding the release of other metals from TFSP.

#### 4.3.3. Burial experiment of oxisol

As shown in Fig. 5c, the patterns of metal concentrations in Oxisol were different from those in synthetic soil and Mollisol (Fig. 5a and b). The Oxisol used in this study was already polluted by various metals, especially Pb. For this circumstance, the initial high pollutant concentrations may suggest that there was ongoing gradual release of pollutants (Fig. 5c). For example, the concentrations of Cu and Zn in Oxisol with only 50 g TFSP added were not significantly different from those in the blank. However the concentrations of Cu and Zn were approximately 29% and 70% higher, respectively, in Oxisol where 200 g of TFSP was added, indicating that the metals released from TFSP caused the metal concentrations to increase in the soil. Compared with synthetic soil and Mollisol, Oxisol showed the highest background concentration of metal contaminant with the lowest pH value. For example, high concentrations of metal contaminants especially of Cu and Pb were obtained. It should be noted that the low pH value can lead to a high leaching efficiency of metal from TFSP, but the results of soil burial experiment figured out the leaching efficiency of Oxisol was much lower than those of synthetic soil and Mollisol. The concentration of Cu increased approximate 29% and the concentration of Pb seemed to be unchanged. It was attributed to the equilibrium of metal contaminants in soil mainly from the background but few from the newly released metals. This was why much lower concentrations of metals were extracted by Oxisol even at a lower pH value. In addition, the concentration of Pb in Oxisol burial experiment decreased slightly, and its concentration did not vary significantly either in the burial experiments of synthetic soil and Mollisol. According to Ma et al. (2007), corroding mechanisms of metallic Pb can be separated into three steps: oxidation, carbonation and dissolution, and soil properties especially pH can significantly influence these processes. This phenomenon possibly reflects differences in the equilibrium status between adsorption and desorption of Pb (including Pb-carbonates and Pb-oxides) and its interaction with other metals. After 60 days of burial, the concentrations of Ni, In and Ga in Oxisol were higher than their initial concentrations increased 114.2%, 89.2% and 492.4%, respectively with 200 g of TFSP being added. The concentration of Cr stabilized at approximately 18 mg/kg. Therefore, the increases in metal concentrations

in these three types of soils reflect the states of metals released from TFSP.

It is noteworthy that, in the same type of soil, the metal concentrations were higher after TFSP was added over the same time interval. In this experiment, by comparing three types of soils after 60 days of burial experiment, the metal concentrations were the lowest in synthetic soil with the highest pH (7.2), followed by Mollisol with a pH value of 5.6, and were the highest in Oxisol with a pH of 3.9, which indicates that pH is one of parameters influences the dissolution of metals in different types of soils, and that metal release from TFSP is enhanced under acidic conditions.

#### 4.4. Mechanism of metal release from TFSP

There are various mechanisms contributed to metal release from TFSP, as follows. First, dissolution and oxidation occurred at the broken interface of TFSP, resulted in metal release into the soil. Second, the ionic strength influenced the rate of metal dissolution, as shown by the variations in dissolution rate after different amounts of TFSP were added related to the background concentrations of metals in three types of soils. Hydrogen ions contributed significantly to the changes in cation exchange capacity in the soil, such that metal release from TFSP was accelerated. Finally, soil properties and surface bonding of soil minerals, for example water molecules, may have facilitated metal adsorption when the  $H^+$  was released, so that metals built up new bonds with oxygen left over on the surface of a mineral (Jiang et al., 2013). The larger amount of hydrogen ion penetrated in the broken TFSP may facilitate corrosion of TFSP, resulting in more metal release into the soil.

#### 4.5. Assessment of soil contamination caused by thin-film solar panel

We used  $P_N$  to assess the degree of metal pollution in soils caused by the metals released from TFSP.

The values of PI for the most abundant metals and the values of  $P_N$  of soil contamination after TFSP was added are shown in Table 3. The synthetic soil, Mollisol, and Oxisol with additions of 200 g of TFSP were classed as heavily contaminated. The  $P_{\text{AVG}}$  showed a strongly positive correlation with addition of crushed TFSP in three types of soils ( $r_{\text{Syn}} = 0.999$ ,  $p < 0.01$ ;  $r_{\text{Mollisol}} = 0.993$ ,  $p < 0.01$ ;  $r_{\text{Oxisol}} = 0.968$ ,  $p < 0.05$ ), indicating that  $P_{\text{AVG}}$  increased with addition of crushed TFSP. This illustrated that more pollutants could be released when more crushed TFSP materials were buried. There was a strong positive relationship between  $P_N$  and addition of crushed TFSP in synthetic soil and Mollisol ( $r_{\text{Syn}} = 0.992$ ,  $p < 0.01$ ;  $r_{\text{Mollisol}} = 0.990$ ,  $p < 0.01$ ), indicating that the  $P_N$  increased as the amount of crushed TFSP increased. This suggests that the contamination levels of synthetic soil and Mollisol increase with the addition of crushed TFSP. The Oxisol with 50 g of TFSP added was lightly polluted, and the lower PI ( $P_{\text{metal}}$ ) for this soil may reflect the fact that it had a higher background of metal concentrations than other soils. The other soils with 50 g of TFSP added were classified as mildly contaminated. The soil samples with TFSP added between 100 and 200 g showed mild, medium, and heavy contaminations, and the contamination level increased as the amount of TFSP added increased. The  $P_N$  values show that TFSP had adverse effects of pollution on the different types of soil.

#### 5. Conclusion

Our results demonstrate that metal pollutants were released into the soil from the buried TFSP. The observations and results show that heavy metals may be released into the surrounding environment once the protective layers are broken and exposed to acidic conditions. We detected 16 metals in TFSPs, of which Ni, Zn, and Cu were released at the highest concentrations in three different types of soils studied. We examined the mechanisms of metal release from TFSP and found that,



from the rates of metal dissolution and the affinity of metals to the reaction media (e.g. soil minerals), they were related to the soil properties. In this study, three various soils showed different abilities to leach pollutants from TFSPs. The synthetic soil showed the best ability for leaching Ni whereas the Mollisol showed the greatest Zn leaching ability. Although the background metal concentrations were highest in the Oxisol, the amount of Cu leached by Oxisol was greatest when the same amount of TFSP was added. The Nemerow Contamination Index showed that all the soils were contaminated to different levels, from light, mild, medium, to heavy, depending on the amounts of TFSPs added. The increases in metal concentrations in the soil were therefore related to the amounts of TFSPs added and the soil properties.

## Declarations of interest

None.

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## Appendix A. Supplementary data

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