Leaching Hazardous Substances out of Photovoltaic Modules

Renate Zapf-Gottwick[1,*], Michael Koch[2], Klaus Fischer[2], Fred Schwerdt[1], Lars Hamann[1], Martin Kranert[2], Jörg W. Metzger[2], and Jürgen H. Werner[1]

[1]*Institute for Photovoltaics, University of Stuttgart, Germany*

[2]*Institute for Sanitary Engineering, Water Quality, and Solid Waste Management, University of Stuttgart, Germany*

Abstract: Photovoltaic modules contain hazardous substances such as lead and cadmium. Under normal operation conditions, these materials will not be released into the environment. This study identifies conditions resulting in release. Our worst case study uses milled module pieces of 0.2 mm size. Depending on the pH value of water based solutions, more or less amounts of hazardous substances are leached out. Solutions with low pH values (acidic solutions) yield substantial leaching. Three different solutions simulate different environmental conditions: i) "low mineralized water" conditions, via water containing sodium hydroxide, ii) "sea water" conditions, via water containing sodium hydroxide and sodium chloride, and iii) "rainwater" conditions, via water containing acetic acid. In "rain water"-like solutions with low pH, already after a few days, around 30 % of the cadmium is leached out from milled cadmium telluride module pieces, increasing to 50 % after 56 days! In the same time, more than 15 % of lead is leached out from c-Si module pieces. Tellurium elutes in the range of 30 to 40 % with a weak dependence on the pH value of the solution indicating an instability of the compound cadmiumtelluride out of the cadmiumtelluride modules. Most of the extractions increase during several weeks of measurement. Therefore, the usual one-day-elution test does not give enough information. Meaningful leaching experiments should last for at least ten days.

Keywords: Cadmium, Elution, Hazardous substances, Lead, pH dependence.

INTRODUCTION

The use of photovoltaic (PV) systems increases in many countries and the PV market growed exponentially in the last years until year 2011. Since 2011 the installation of modules has changed to a linear growth. Nevertheless, in the year 2014, the total world wide installed capacity reached 177 GW_p. The price reduction of PV modules drives now the market. This PV power corresponds to about 16 million tons of PV modules. With a forecast for the installation of 604 GW_p in the year 2020, the installed weight of modules will increase to 47 million tons [1].

Photovoltaic modules are "green products": They do not only produce environmentally friendly electricity, but also their production (if properly carried out) has minor influence on the environment. Unfortunately (and basically unnecessary), most of the photovoltaic modules contain hazardous substances: On the one hand, even if the solar cells themselves are free of toxic elements or compounds (as is nearly the case with modules from either crystalline, microcrystalline or amorphous silicon) the *soldered ribbons* within the modules contain the toxic heavy metal lead, Pb. The same holds also for the thin film modules made out of cadmium sulfide/cadmium telluride, CdS/CdTe, and

copper indium gallium diselenide, $Cu(In,Ga)Se_2$ (CIGS). On the other hand, in case of CdS/CdTe modules, the semi conductors themselves are toxic. In case of CIGS modules, the compound $Cu(In,Ga)Se_2$ seems less toxic, however, many manufacturers of such modules use the toxic compound CdS as part of the window layer. In fact, only few companies produce really "green" modules, being completely free of heavy metals such as Pb and Cd [2].

Despite containing toxic substances, under "normal" operation conditions, PV modules seem not to impose any ecological danger on the environment. However, even under "normal" conditions, it is possible that water penetrates into modules either via cracks (for example, induced by improper installation or by mechanical damage such as by hail) in the module glass, through the back sheet, or via a defective lamination. Clearly, under such conditions, there is a chance that heavy metals are leached out. These cases seem rare, however, they exist! Over an assumed operation of 20 years, probably several percent of the modules will fall under this category. Even worse, however, it is unclear at present what happens to all the modules at the end of their "lifetime", which is usually assumed to be 20 to 25 years. If such modules are not collected and properly recycled, they might either be dumped somewhere or, simply be "donated", or (legally or illegally) exported to countries, which do not have clear regulations on garbage collection and recycling. Even if

[*]*Address correspondence to this author at the Institute for Photovoltaics, University of Stuttgart, Germany; Tel: +49 711 68569 225; Fax: +49 711 68567 143; E-mail: Renate.Zapf-Gottwick@ipv.uni-stuttgart.de*

such „donations" or „exports" were or will be illegal, they will probably occur, as is the case for „normal" electronic garbage. Thus, it remains unclear what will happen to the toxic materials in the PV modules.

Toxic substances in PV modules have been under political and scientific debate for several years. Several publications investigated if toxic materials are able to leave the modules by emission during production, due to fire, or by leaching in aqueous solutions [3-8]. Unfortunately, these investigations show contradictory results in leaching hazardous substances out of modules [3-7]. However, these investigations were also not coherent: They differ in the kind of aquaeous solutions used for leaching, in the size of module pieces to be leached out, in the investigated module technology, and, finally, also in the testing procedures (*e.g.* time of leaching).

The present contribution uses worst case conditions for the leaching of photovoltaic modules in aquaeous solutions with different pH values. To accelerate the process of leaching, we mill down module pieces to a size of 0.2 mm. The milled module pieces are extracted and shaken in water-based solutions for a time of around one to two months. We use pH values in the range $3.4 \leq pH \leq 8.4$. Choosing neutral water with pH ≈ 7 suppresses leaching of Cd and Pb. Under these specially chosen conditions, our study completely confirms the literature data of leaching experiments on CdTe [3]. However, in contrast to previous work [3,5,7], we did not only leach out for one day, but for several weeks. In this respect, our studies are much more realistic, when it comes to simulate the behavior of broken modules, which might be (legally or illegally) dumped in landfills, for example, where they remain

forever. Clearly, such modules will unavoidably not only be leached out by water, they are also attacked by organic acids and bio organisms. Thus, it is not a question if they are leached out, but only how long does it take? Our leaching study reveals that leaching of hazardous substances out of PV modules depends mainly on the pH of the aquaous solutions.

MATERIALS AND METHODS

1. Sample Preparation

We mill module pieces of the four major commercial technologies: i) crystalline silicon (c-Si), ii) cadmium telluride (CdTe), iii) copper indium gallium diselenide (CIGS), and iv) amorphous silicon (a-Si). The modules are typical for their technology and bought on the open market. In contrast to the thin film modules, the c-Si module has only one glass plate. Figures **1a** and **b** show the module preparation: In order to get a defined and homogenous amount of module material, we cut a commercial module into pieces of 7x7 cm^2 size (Figure **1a**). These pieces do not contain any parts from the metallic frame or material from the module box or cables and are taken from the part with photovoltaic active material. In the case of thin film modules, the cut pieces contain parts of the ribbons, which connect the cells to the module box. Water jet cutting helps to get defined pieces with homogenously cut edges; otherwise the tempered module glass will split. After cutting the module pieces, they are milled to grain sizes of less than 0.2 mm. For every experiment, we dissolve exactly 100 g of the milled module powder in the three different aqueous solutions, given in section 2, with a volume of two liters each. Then, as shown in Figure **1b**, we shake every solution overhead for several weeks.
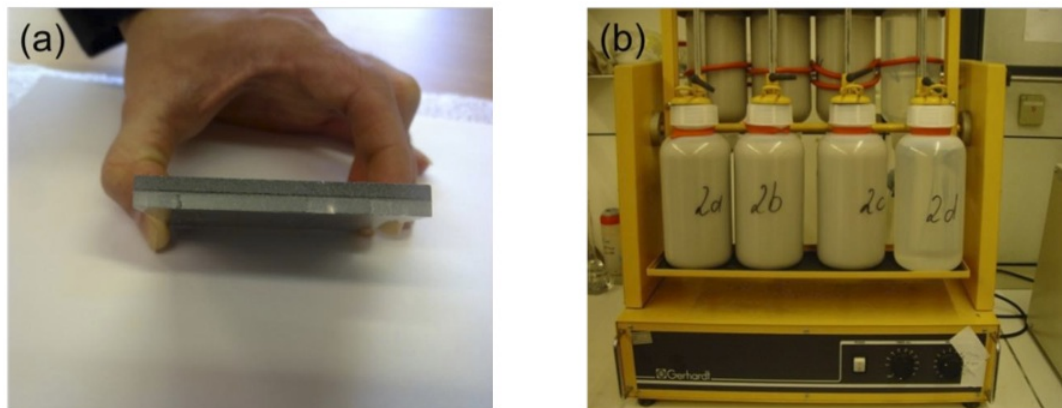

Figure 1: Sample preparation: **a**) active part of a photovoltaic module without frame or module box is cut into pieces by water jet cutting. **b**) After milling to grain sizes of 0.2 mm, the milled module powder is extracted in aqueous solutions. The bottles with solutions inside are shaken overhead for several weeks.

2. Leaching Solutions and Sampling

The leaching experiments are carried out at room temperature. To simulate different environmental conditions, we use the following three water based solutions:

1.   LMW: "Low mineralized water", simulated by 200 mg $NaHCO_3$ dissolved in 1 liter of water, reaching $pH_{LMW} \approx 8.4$.

2.   SW: "Seawater", simulated by 1 g $NaHCO_3$ and 30 g $NaCl$ dissolved in 1 liter of water, reaching $pH_{SW} \approx 7.8$.

3.   RW: "Rainwater", simulated by 0.1 % acetic acid with $pH_{RW} \approx 3$.

Our test solutions are representative for realistic environmental conditions. The pH value of rainwater and landfills differs with location and conditions: In Brazil the pH of rainwater ranges at $4.2 < pH < 6.1$ [9], whereas in Germany the pH value is higher with $4.1 < pH < 5.3$ [10]. Landfills in Germany range at $3.4 < pH < 11.9$ depending on the classification of waste [11], while seawater has pH values of $7.5 < pH < 8.5$ [12], more in the range of neutral water, which has $pH = 7$. Mineralized water, depending on its origin, also varies in the range of $4.9 < pH < 8.4$ [13].

Samples of the solutions for analyzing the leached elements are taken at the beginning of the experiments and then in time intervals of several days. With every sampling, the content of the leaching solution is corrected by the amount of solution and leached elements which are extracted. Inductively coupled plasma optical emission spectroscopy (ICP-OES), and atom absorption spectroscopy (AAS) after electrothermal atomization in a graphite furnace analyze the amount of leached substances. The measurement uncertainty is around +/-10 %. The total content of the elements is determined by ICP-OES and AAS after oxidative digestion. Organics and organic compounds with metals oxidize to carbon dioxide and water, not troubling the measurement of metals.

3. MATERIAL CONTENT OF THE MODULES

After oxidative digestion of a part of the powder, we analyze the total content of the relevant elements per kg of module mass M_{mod}. Table **1** gives the calculated power related mass G_X for every element X (X = Pb, Cd, Te, Se, Ag, In, Ga) of the elements found in the four different module technologies: c-Si, CdTe, CIGS, and a-Si. The measured content of the elements is related to the module power P_{mod}, respectively the mass M_{mod} of the modules. In addition to the toxic elements Pb, Cd, Te, Se, we also give values for the rare materials Ag, In, Ga. According to Table **1**, we find Pb in every module, up to $G_{Pb} = 80$ mg/W for the c-Si modules. The reason lies in the lead containing solder of the copper (Cu) ribbons. In the c-Si modules, these ribbons are used for series connecting every single cell in the module. In addition, the ribbons connect the cell strings to the module box. The thin film technologies use an integrated series connection between neighboring cells. However, even in their case, we find a substantial amount of Pb. This Pb stems from the ribbons connecting the first and last cell in the module with the module box. Table **1** shows that even a-Si modules contain Pb, simply due to these ribbons. In c-Si modules, the amount of Pb is highest because of the larger number of ribbons interconnecting the solar cells. Usually, the solder of the Cu ribbons contains around 28 % of Pb [14]. A relatively small part of Pb stems from the metallization pastes of the solar cells with 0.5 to 1 mass per cent. The front side and the back side contact, contain not only Ag and Al but also oxides from Pb [15,16] and sometimes from Cd, Zn, Th, Bi combined under the name of "glass frit" [17,18]. Due to Cd-oxides in the screen printing paste, we also

Table 1: Power P_{mod} and mass M_{mod} of the Modules as well as the Measured Total, Power Related Mass G_X of Element X (X=Pb, Cd., Te, Se, Ag, In, Ga) in the Leached PV-Modules.

Module	P_{mod} [W]	M_{mod} [kg]	G_{Pb} [mg/W]	G_{Cd} [mg/W]	G_{Te} [mg/W]	G_{Se} [mg/W]	G_{Ag} [mg/W]	G_{In} [mg/W]	G_{Ga} [mg/W]
c-Si	170	14.8	80.2	0.22			75		
CdTe	72	11.8	17.7	73	94				
CIGS	80	12.6	12.9	1.9		52		21.6	7.2
a-Si	100	18	4.3				2.6		

find trace amounts of Cd in c-Si modules. Clearly, CdTe-modules contain the largest amount of Cd with G_{Cd} = 73 mg/W, which stems from the several μm thick CdTe layer as well as from the thin CdS window layer forming the CdS/CdTe heterojunction. In case of CIGS modules, the Cd stems from the about 50 nm thick CdS in the window layer. Tellurium, only found in CdTe modules, stems from the CdTe layer. In case of the c-Si modules, the G_{Ag} = 75 mg/W silver stem from the screen printing paste for the front contacts to the n-type emitter and from the backside busbars. In case of the a-Si modules, the much less G_{Ag} = 2.6 mg/W stem from the back contacts to the individual a-Si cells. Clearly, in case of CIGS-modules, we must find In and Ga in the ratio 3:1, which is typical for high efficiency CIGS cells [19].

RESULTS

This section shows the results of leached out elements with respect to the total amount of elements being contained in the modules given in Table **1**. The experiments run over 28 to 56 days, depending on the development and progress in leaching of the elements. Only the elements in solution are analyzed, precipitations in the solution are not measured as leached. Leached metal ions in saline solutions (like SW) form chlorides and precipitate as solid matter. They are not included in the results. The pH-value of the solutions is measured at the beginning (pH_{start}) and at the end of the experiment (pH_{end}). Due to the milled glass particles (which contain metal oxides) of the modules, in all leaching experiments, the pH increases during the experiments depending on pH_{start} of the solution with a difference $\Delta pH = pH_{end} - pH_{start} \approx +3$. Experiments are carried out twice or three times

represented in the figures by different symbols in the same color.

1. c-Si Modules

Figure 2 shows the time dependence of the leaching of Pb and Ag out of milled c-Si modules in RW and LMW solutions; Pb and Ag in SW are not measured due to their precipitation as chlorides. In Figure **2a** the elution of Pb in RW and in LMW starts with $Pb_{c-Si} \leq 0.2$ %. In LMW it remains low, whereas in RW Pb_{c-Si} increases to $Pb_{c-Si} = 40$ % already after a time $t = 10$ days. Figure **2b** shows a low leaching of Ag in RW and LMW with $Ag_{c-Si} \leq 1$ %. The RW-solution starts with $pH_{start} = 3.2$. After 42 days of leaching the pH increased to $pH_{end} = 6.1$ which explains the decrease in Pb_{c-Si} and Ag_{c-Si}. The LMW-solution shows also an increase in pH from $pH_{start} = 8.6$ to $pH_{end} = 11.3$.

2. CdTe Modules

Figure **3a** shows the increasing amount of eluted Cd_{CdTe} and Figure **3b** eluted Te_{CdTe} out of milled CdTe modules. The elution of Cd_{CdTe} is low in LMW and in SW because of a precipitation of cadmium carbonates or hydroxides. In RW, Cd_{CdTe} is highest, leading to $Cd_{CdTe} \approx 50$ % after $t = 56$ days. In contrast, Te_{CdTe} is eluted in the range of 30 % $< Te_{CdTe} <$ 40 % in every aqueous solution. Independent of the pH of the solution, the bonding of CdTe decomposes. The higher the pH value, the higher is the formation of insoluble cadmium hydroxide or carbonate, explaining the lower amount of measured Cd_{CdTe} compared to Te_{CdTe}. Starting the extraction of the milled CdTe-modules, the pH increases in every solution: in RW from $pH_{start} = 3.1$
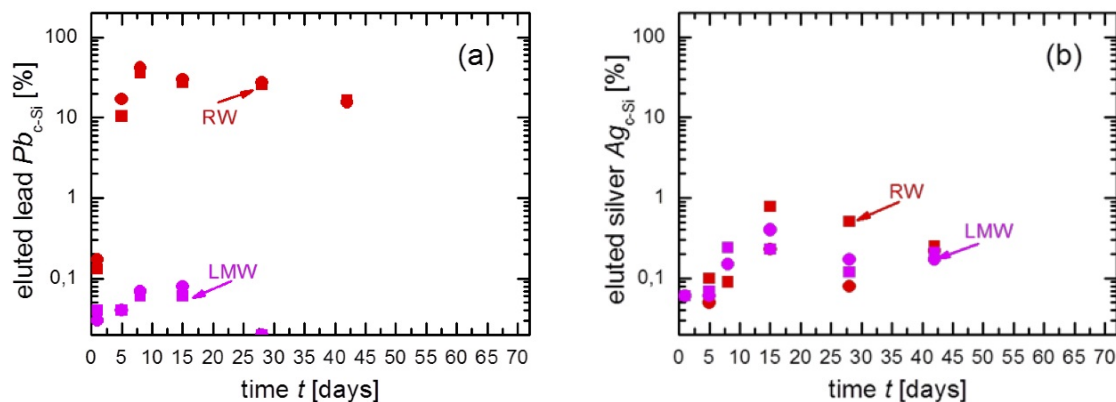

Figure 2: Leaching of Pb_{c-Si} and Ag_{c-Si} out of milled c-Si modules. Pb and Ag in SW precipitate as chlorides which are not measurable in solution. a) Eluted Pb reaches $Pb_{c-Si} = 40$ % after t = 10 days in RW, in LMW $Pb_{c-Si} \leq 0.1$ %. b) Less than 1 % of Ag_{c-Si} is eluted in RW as well as in LMW. In LMW, the pH changes during the extraction from $pH_{start} = 8.6$ to $pH_{end} = 11.3$, in the RW from $pH_{start} = 3.2$ to $pH_{end} = 6.1$.
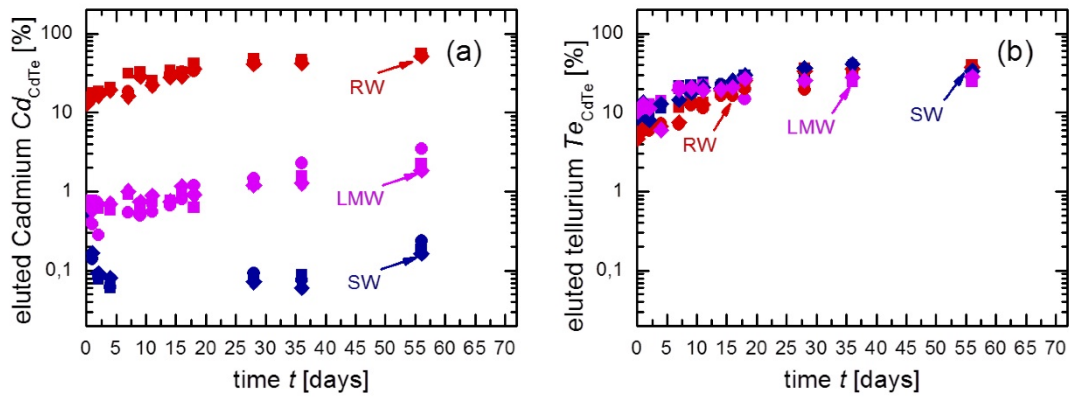
Figure 3: Elution of Cd_{CdTe} and Te_{CdTe} out of milled CdTe modules. a) Cd_{CdTe} increases in RW after t = 56 days to $Cd_{CdTe} \approx 50$ %, in LMW only in the range of single-digit, and in SW with $Cd_{CdTe} < 1$ %, but increasing. b) Eluted Te_{CdTe} increases to a saturation value $Te_{CdTe} \approx 40$ % after t = 56 days, independent of the kind of solution. In LMW pH changes during extraction from pH_{start} = 8.4 to pH_{end} = 11, in SW from pH_{start} = 7.8 to pH_{end} = 9.5, and in RW from pH_{start} = 3.1 to pH_{end} = 5.

to pH_{end} = 5, in SW from pH_{start} = 7.8 to pH_{end} = 9.5, and in LMW from pH_{start} = 8.4 to pH_{end} = 11.

3. CIGS Modules

The active layer in CIGS modules contains the elements Cu, In, Ga, and Se. The element Cd is found in the CdS-layer, and Zn, as a part of the transparent conductive zinc oxide ZnO, is also measured. Figure **4a** shows the eluted amount of Cd_{CIGS}, and Figure **4b** the eluted amount of Se_{CIGS}. The extraction of Cd_{CIGS} out of CdS behaves like the extraction out of CdTe: at most in RW with $Cd_{CIGS} \approx 30$ %, less in LMW and SW. The mobilization of Se in alkaline solutions, like SW, is higher than in RW, and maximal in SW with $Se_{CIGS} \approx 10$ %.

Figures **5a** and **b** present the extraction of Indium and Ga. Independent of the kind of solution and its pH, the elutions of In and Ga are less than In_{CIGS}, $Ga_{CIGS} <$

1 %. In SW, the elements In as well as Ga form insoluble precipitations.

The elution of Zn is high with $Zn_{CIGS} \approx 50$ % out of the ZnO-layer in RW, in LMW, it holds $Zn_{CIGS} < 1$ %. The extraction, as Figure **6a** shows, does not increase in t = 28 days. In contrast, the eluted Cu_{CIGS} increases with time in RW, but in LMW remains constant to $Cu_{CIGS} \approx 0.1$ %, as seen in Figure **6b**. Both elements Zn and Cu precipitate in SW.

With the start of the extraction of the milled CIGS module the pH rises, in LMW from pH_{start} = 7.6 to pH_{end} = 10.5, inSW pH from pH_{start} = 7.0 to pH_{end} = 10.3, and in RW from pH_{start} = 4.3 to pH_{end} = 4.9.

4. a-Si Modules

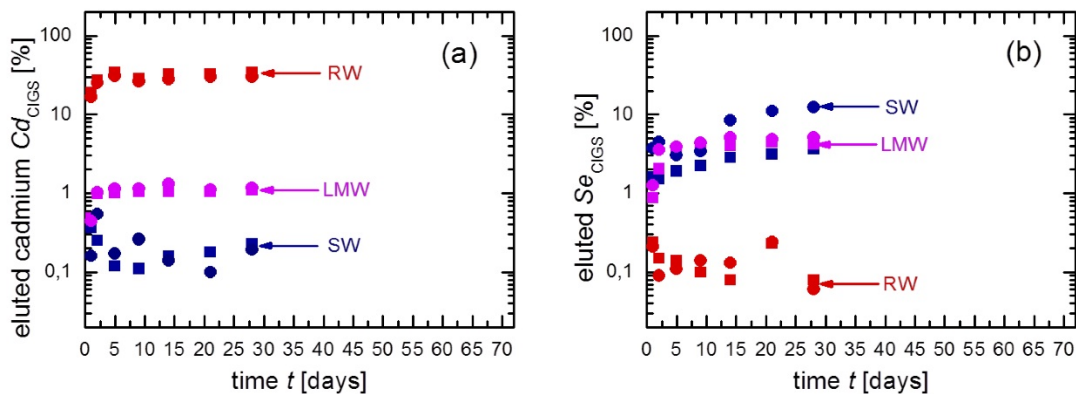In case of these modules, the active layer itself does not contain any hazardous substances. However,

Figure 4: Elution of Cd_{CIGS} and Se_{CIGS} out of milled CIGS modules. **a)** Extraction of Cd increases to $Cd_{CIGS} \approx 30$ % in RW, in LMW in the range of single-digit, and in SW with $Cd_{CIGS} < 1$ %. **b)** Extraction of Se in SW rises to $Se_{CIGS} \approx 10$ %, in RW only $Se_{CIGS} < 1$ %. In LMW the pH changes during extraction from pH_{start} = 7.6 to pH_{end} = 10.5, in SW from pH_{start} = 7.0 to pH_{end} = 10.3, and in RW from pH_{start} = 4.3 to pH_{end} = 4.9.
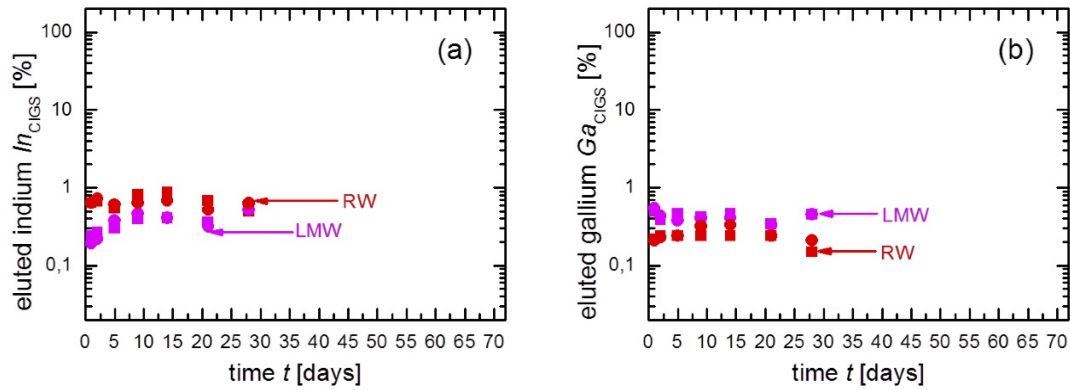
Figure 5: Elution of In_{CIGS} and Ga_{CIGS} out of milled CIGS modules. a) Extraction of In_{CIGS} in RW and in LMW rests $In_{CIGS} < 1$ %. b) Extraction of Ga remains at $Ga_{CIGS} < 1$ %. In SW the elements in and Ga form insoluble precipitations. For change in pH see caption Figure **4**.
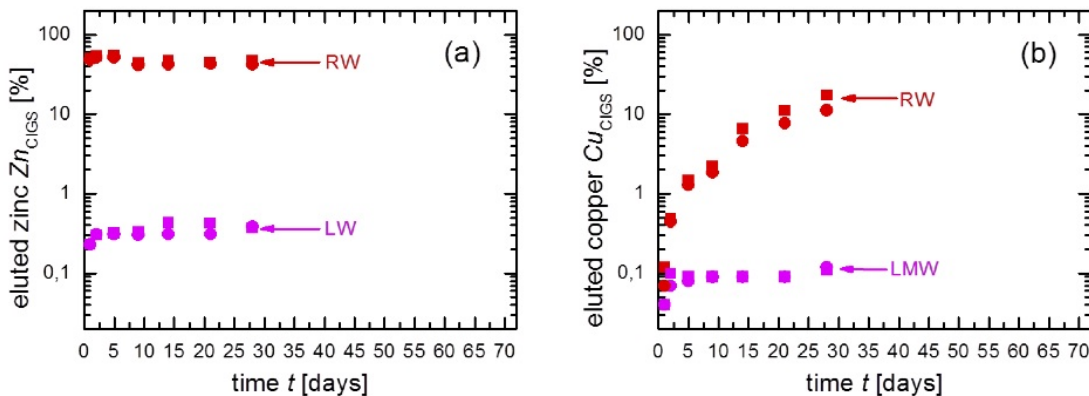

Figure 6: Elution of Zn_{CIGS} and Cu_{CIGS} out of milled CIGS modules. **a**) 50 % of Zn is extracted in RW, in LMW in the range of less than 1 %. **b**) Eluted Cu in LMW lies in the 0.1 % range, in RW increasing with time, after 28 days to 16 %. For change in pH see caption Figure **4**.

a small amount of Pb and Ag is used in the electrical connections. Our leaching experiments detect only low amounts of eluted Pb and Ag, close to the detection limit. As in all other experiments, the pH increases during the experiments in every aqueous solution with $\Delta pH \approx +3$. At present the low leaching of Pb and Ag is not understood. Experiments are going on to solve this issue.

DISCUSSION

So far, only a few leaching studies are available. In most cases, only CdTe-modules were investigated; leaching experiments on other PV-modules are scarce. The previous leaching studies on CdTe-modules yielded contradictory results. The experiments used different conditions: Mainly the size of the module pieces, the solutions, the pH-value (often not given, [20]), and also the leaching times differ [3,5,6]. Therefore, at first sight, a comparison seems difficult.

However, here we show that the pH value is the critical parameter: Table **2** compiles the parameters of five different, previous studies. We reference the studies and list the material, size of module pieces, and leaching time.

Figure **7** collects the data from the viewpoint of the pH-value of the solutions; our own data fit perfectly into the schema. For our own data, we use the pH-value pH_{end} and the final amount of leached elements of our time dependent experiments. The numbers in Table **2** correspond to the numbers in the data points of Figure **7**. It becomes obvious, that not the size of the module pieces, but the pH value plays the major role: The more acidic the solution, the more Cd and Pb is leached out! Only Te leaching shows a weak dependence on pH. In total, about 30 % of the Te is leached out, indicating that the compound CdTe is unstable under all conditions. In particular, as soon as the solution is acidic, the toxic element Cd is more or less completely

leached out, whereas no leaching is visible for pH > 7. In this case Cd precipitates in alkaline solutions and is no longer dissolved. Our results compare well to earlier "availability tests" of Refs. [5,7] where crushed module pieces with a size of 125 µm were leached in aqueous solutions. In their [5,7] case, the pH was decreased from pH = 7.7 to pH = 3.2 by addition of nitric acid and sulfuric acid: The lower the pH, the higher is the leaching of Cd out of crushed CdTe modules and also of Pb out of c-Si modules.

The almost complete leaching of Cd and Pb under acidic conditions with pH < 7 appear severe: Such conditions do appear in rain water [9,10]. Due to carbon dioxide intake from the atmosphere, rainwater is always acidic with a typical pH ≈ 5.
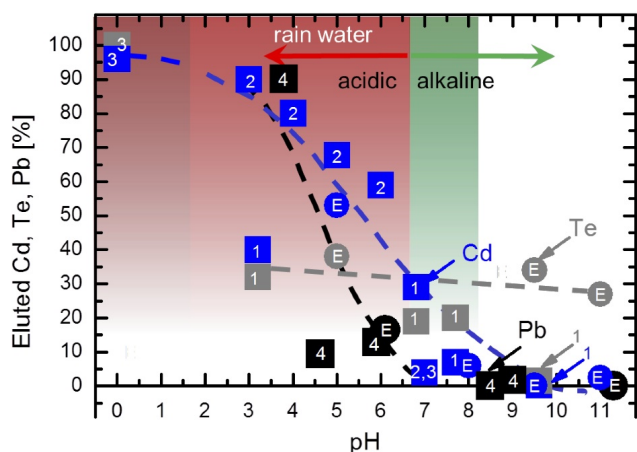

Figure 7: Eluted Cd, Te, and Pb in solutions with different pH. The data of own experiments are marked with "E"; we took the data of pH and eluted amount of element at the end of our experiments. Literature data, marked with numbers from 1 to 4, stem from Table **2**. Dashed lines are guides to the eye. Leaching of Cd and Pb highly depends on the pH highly increasing in the acidic range (regime of "rain water"). In contrast, Te leaching depends only weakly on pH in the range 3 < pH < 10.5.

CONCLUSIONS

Our leaching experiments have demonstrated severe leaching of Pb from c-Si modules and Cd from CdTe modules. In addition, our leaching studies on CdTe modules prove that CdTe is not stable in water based solutions. More than 50 % Cd is leached out within 56 days for pH = 5. In the same time, Te leaches in the range of 30 %, more or less independent from pH, whereas more than 15 % Pb is leached out for pH = 6. In our case, the modules are milled to pieces of 0.2 mm size. Leaching time was several weeks to get information about the time dependence. Disagreements and contradictions in previous studies are solved by considering the pH value of the solutions.

So far, the discussion about leaching of hazardous substances mainly concentrated on the element Cd. However, the 80 mg/W content of Pb in c-Si modules, as well as the 75 mg/W silver come close to the 73 mg/W of Cd! Thus, if one discusses toxic materials in PV materials, there is not only a problem in case of CdTe modules but also in case of c-Si! One should also bear in mind that the market share of c-Si modules (≈ 90 %) is much larger than the one of CdTe modules (about 5 %). On the basis of a worldwide installation of 177 GW PV modules in 2014, the c-Si modules with around 158 GW containing 80 mg/W Pb distributed 12800 t of Pb over large areas! In case of CdTe modules with an estimated installation of 8.3 GW, the CdTe modules distributed only 610 t of Cd. Even assuming that Pb is much less toxic than Cd, one must admit that the Pb problem in photovoltaics is much more severe than the one of Cd. From this viewpoint, it is hard to understand why Pb containing solders are still used in PV; lead-free soldering has been a standard in electronic industry for many years. In addition, the c-Si modules of several Japanese producers are completely free of lead!

Table 2: Previous Leaching Studies on CdTe- and c-Si-Modules. Size of Module Pieces, Leaching Time and pH of the Leaching Solution is given; Eluted Elements and Corresponding pH are Plotted in Figure 7.

No.	Year	Ref.	Author	Laboratory	Module	Size	Time
1	2010	[5]	Okkenhaug	Norwegian Geotechnical Institute	CdTe	125 µm < 4 mm	< 1 day 1 day
2	2010	[6]	Allen, Kiss	Amelio Solar	CdTe	5x5 cm^2	200 days
3	2006	[3]	Fthenakis	Brookhaven Nat. Lab.	CdTe	hammer milled	4 hours
4	2010	[7]	Arp	Norwegian Geotechnical Institute	c-Si	125 µm < 4 mm	< 1 day 1 day

Leaching of hazardous substances from photovoltaic modules is real. In particular, our investigations raise serious questions about the leaching in normal, "neutral" rain water or in landfills, which have pH < 7. Photovoltaic modules have a high risk potential by releasing hazardous substances to the environment if they are not properly (legally or illegally) disposed in domestic waste landfills, in building rubble dumps, or on wild waste dumps as might be the case in developing countries. Toxic materials, such as Cd and Pb will be leached out completely. It is only a question of time!

ACKNOWLEDGEMENT

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