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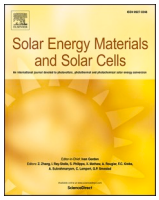

Selective recovery of the CIGS and Mo layers from thin film solar cells

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ABSTRACT

The Cupper Indium Gallium diSelenide (CIGS) Photovoltaic (PV) technology is well established in the market and considered as a good candidate for incorporation in other, more recently developed and complex PV technologies (e.g. tandem). Despite its popularity, little research has been done on the recycling of its materials, which contain valuable, critical and toxic elements. In this paper, the possibility of recovering in solid form the valuable CIGS material from CIGS solar cells, through first selectively leaching the Mo layer deposited underneath and subsequently liberating the unsupported CIGS by mechanical means, was explored. Different NaOH concentrations, process temperatures and means of mechanical liberation of the unsupported CIGS were investigated. The results showed that although a range of the tested NaOH concentrations could efficiently dissolve the Mo even at ambient temperature, for 0.1-0.5 M NaOH, some of the CIGS reacted with the NaOH and the compositional elements of the former ended up scattered in various phases. This was not the case when using a dilute NaOH solution of pH = 11 at 50 °C with an automatic titration system for keeping the pH constant. More specifically, after the application of these conditions for 8 h and a subsequent mechanical brushing of the substrate, the composition and crystalline structure of the recovered CIGS had remained practically unaffected, compared with the untreated material. Complete recovery of the CIGS was then achieved, with a purity of about 95 wt%. Simultaneously, 82 wt% of the Mo was recovered in the leachate.

1. Introduction

Although the crystalline-silicon (c-Si) PV technology dominates the market today, it is the thin-film PV technology that offers the advantage of a similar efficiency [1] but with a much lower need in raw materials. The market share of the CIGS thin film PV is about 1% [1], but the technology is also incorporated in the fast-developing tandem PVs [2]. As the world-wide electricity production generated by all PV technologies is expected to reach 25% in 2050 [3], a subsequent increase in the amount of PV waste, including the ones coming from the CIGS technology, should also be expected. Waste does not only originate from end-of-life PV, but also from the PV manufacturing process, therefore, the need for efficient, sustainable and attractive for the industry waste recycling processes becomes apparent.

The unit which converts the sunlight into electricity in a PV system is the solar cell. A CIGS solar cell consists of multiple thin film layers deposited on top of each other, with the most important of them being the following [4–8]: The base material is usually a substrate made of glass, plastic or stainless-steel, on top of which a layer of Mo is deposited, as the back contact. Then, the CIGS light photon absorber film follows. The buffer layer, commonly CdS or one of the ZnS, ZnSe, In_2S_3, Zn(S, O, OH) and Zn(Mg)O, is deposited on top of the CIGS film, followed by the window layer, usually ZnO. The last and topmost film layer is the Transparent Conductive Oxide (TCO) layer, made of Indium Tin Oxide (ITO) or ZnO:Al (AZO). As soon as the deposition of all film layers is completed, the conductive Ag or Al/Ni grid is finally added on top of the cell. All layer thicknesses are in the range of nm to few μm [4–8], therefore, the concentration of any valuable element per cell mass is very low. It becomes apparent that a plethora of elements can be present in a CIGS solar cell, with many of them being valuable or critical, for example, Ag, Cu, Ga and In [9,10]. Other elements, like Mo, are of high importance for today's economy [10]. In the case of Se, this is a toxic element and can cause serious health issues, if not treated properly [11]. Therefore, responsible waste management of CIGS-containing waste is necessary for economic, environmental and health-related reasons.

Because of the low elemental concentrations present in the CIGS

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solar cells, waste treatment methods based on hydrometallurgy are preferable over pyrometallurgical methods, as the former offer a more efficient control and tuning of individual waste streams. However, one may say that the industrial processes for recycling PV materials are still in their infancy, with much research yet to be done: The available papers on the topic are very few and their majority deals with the recovery of Cu, Ga, In and Se from CIGS-containing production waste (e.g. chamber waste and spent sputtering targets), while only a small minority deals with whole PV modules and panels, whose recycling is more challenging, due to the presence of an increased number of materials and elements and the low concentrations of the valuable metals. When whole modules or panels are recycled, the organic layers (encapsulants) between which the solar cells are sandwiched should first be opened, in order to leave the metals of the solar cells exposed to further treatment. This can take place by shredding the module, using liquid N_2 for separation of the layers with different thermal expansion coefficients, lasers for evaporation of the back contact and thus detachment of the cell layers from the substrate etc [12]. As soon as the metals are exposed (regardless of the initial source of the CIGS-containing waste), the proposed recycling methods usually start with a high temperature step for recovering the Se as volatile SeO_2 [13–15] and a leaching step for dissolving the Cu, Ga and In, using fairly concentrated solutions of mineral acids and elevated temperatures [13,14,16]. Separation and purification of the individual elements, commonly using solvent extraction [13,16] and/or precipitation [13,14,16], often follow as the final steps. Although the suggested recovery routes seem to be efficient, their main disadvantage is the relatively high consumption of chemicals and energy, as well as the involvement of toxic gases. Therefore, the possibility to leach CIGS solar cells with solutions of relatively low HNO_3 concentrations ($\leq$2M) at ambient temperature was recently explored with promising results [8]. However, the main drawback of this method was its non-selectivity [8], which can increase the needs for further separation and purification of the elements in subsequent steps. To the best of our knowledge, the only method developed so far for selective separation of the CIGS layer coming from a cell or module is the one suggested by Kushiya et al. [17], which used a metal scriber and a vacuum dust remover, in order to liberate the CIGS from the solar cell and recover it in solid form. As this method uses only mechanical means for the separation, no chemicals are involved in the process and no high temperature is used. However, contamination of the CIGS material from any material underneath it is likely, as the latter can also be scribed along with the CIGS.

Notably, the available literature on the recovery of materials from CIGS PV cells has so far only focused on the four CIGS compositional elements and Ag [8,18]. Despite the importance of Mo for the modern economy and its widespread industrial use [10], a separation or recovery of the element from PV waste has largely been neglected. To the best of our knowledge, the only researchers who have touched upon the matter are Kushiya et al. [17] and Teknetzi et al. [8], who have shown that the Mo from CIGS solar modules can dissolve in HNO_3, under specific conditions. However, as previously explained, mineral acids are commonly used for the leaching of the CIGS material too, resulting in non-selective leaching of Mo and, thus, simultaneous losses of the valuable CIGS [8]. Any other literature found on the dissolution of Mo compounds regards only Mo-containing spent catalysts [19,20] and steel making dust [21]. It is worth noting that the Mo found in these waste sources is not in its metallic form (in contrast to most of the Mo in CIGS solar cells), but either in the form of sulphides [19] or oxides [21], which were treated with alkaline leaching [19,21], or in the form of Na_2MoO_4, which facilitated hot water leaching [20]. Solvent extraction [19,20] or precipitation [21] have been suggested for Mo separation from the alkaline leachates. To conclude, alkaline leaching seemed worth investigating for selective leaching of Mo from CIGS solar cells.

In this article, the possibility of selectively recovering in particulate form the CIGS material coming from CIGS solar cells has been explored. This could enable the further treatment of the CIGS, by providing a raw material with reduced initial contamination compared to the methods suggested so far, so that further purification becomes easier. Moreover, having the recovered CIGS in particulate form means that the material can be handled as a powder from now on, so feeding it in existing reactors and creating solutions more concentrated in metals should be easier than when handling solar cell pieces. Finally, the selective recovery of the CIGS as solid particles can also open the possibility to test its reuse (after proper purification to 5N, required for CIGS solar cell [15]), i.e. without dissolving it first and resynthesizing it.

Before the experiments took place, the ZnO, ITO and Ag present in the cell had already been removed by applying a 2-step ultrasonic (US)-leaching process, described elsewhere [18]. The selective separation of the CIGS was attempted in the current paper through applying alkaline leaching first, which targeted the selective dissolution of the Mo layer. As a result, the unleached CIGS layer, which would remain unsupported after the dissolution of the underlying Mo layer, could easily get liberated by mechanical means in a subsequent step and recovered in solid form. The effect of the concentration of the leaching agent (NaOH) and temperature on the efficiency and selectivity of the method were investigated, as well as methods for liberating the solid CIGS material from the leached substrate. The successfulness of the various tested methods was evaluated by analyzing the leachate's elemental composition and characterizing the recovered CIGS particles and comparing them with the untreated CIGS. The investigated approach can offer not only the advantage of recovering the valuable CIGS material selectively, but also recovering the important for today's economy Mo. Moreover, after a successful removal of the film layers, the remaining stainless-steel substrate can easily be recycled industrially, along with other steel waste, closing in this way the material loop.

2. Theory

As previously mentioned, in the case of Mo in CIGS solar cells, the element is mainly present in its metallic form. Alkaline leaching is not recommended in the literature for metallic Mo dissolution [22], although it is not clear if the recommendation also applies to small amounts of the element, as in our case (i.e. a bit more than 60 mg Mo/cell, meaning about 100 ppm Mo in the leachate if each cell is leached with about 720 ml solution, volume suitable for achieving high Mo dissolution efficiency, at least for acid leaching [8]). Instead, the literature recommends the use of alkaline solutions for the dissolution of MoO_3 towards formation of molybdates (MoO_4^{2-}) [22]. A direct conversion of Mo to MoO_4^{2-} could be beneficial though: Provided that the direct conversion does not require harsh conditions, the fewer the processing steps, i) the lower the total environmental impact of recycling, ii) the lower the likelihood for the CIGS material to undergo changes and losses from one step to the other and iii) the simpler the total recycling process, which can be attractive for scale up. Therefore, this possibility was explored further.

To explore the possibility of Mo dissolution under alkaline conditions, the net reaction of Mo with NaOH was simulated with the software HSC:

$$\mathrm{Mo(s) + 2NaOH(aq) + 2H_2O \rightleftharpoons Na_2MoO_4(aq) + 3H_2(g)}, \quad \Delta G^0_{20^\circ C} = -117\ \mathrm{kJ/mol\ Mo} \tag{1}$$

In this system, Mo is oxidized to Mo^{6+} by H_2O, which is reduced to H_2(g). The considerably negative ΔG° of the net reaction (1) for a wide range of temperatures (Fig. S1a, Supplementary material) indicates that the reaction could be expected to be spontaneous. Some more information regarding the predicted leaching reaction of metallic Mo with alkaline solutions, for specifically the concentration of Mo present in our experiments, was obtained from its Pourbaix diagram, constructed with the HSC (Fig. S2a, Supplementary material). These plots showed that, when pH > 9, the metallic Mo is expected to first oxidize to MoO_2 for a narrow range of electrochemical potential and, then, convert to MoO_4^{2-},

as soon as the potential of the solution increases further. The kinetics of the reaction though had to be tested experimentally.

The other form in which Mo is found in CIGS solar cells is that of $MoSe_2$, which is formed as an interface layer between the CIGS and the metallic Mo during the manufacturing of the cell. This layer is usually around 100-200 nm (a few times thinner than the metallic Mo layer) [23]. It is known that $MoSe_2$ can act as a catalyst for hydrogen evolution, both under acidic as well as alkaline conditions [23,24]. Although catalysts are not expected to be consumed, some researchers have reported dissolution of $MoSe_2$ in the process of hydrogen evolution in alkaline solutions, towards formation of MoO_4^{2-} and SeO^{2-}, which can later convert further to MoO_7^{2-} and SeO_4^{2-} [25].

The thermodynamics of the leaching of the rest of the elements which were present in the solar cells used in the particular paper, i.e. Cu, Ga, In, Se, Ti and W, was important to be considered as well, in order to assist with the selection of suitable experimental conditions. It is worth clarifying here that although W and Ti are two elements which are usually not mentioned in general descriptions of a CIGS solar cell, they can be used as an alloy barrier for stainless-steel substrates, in order to hinder the diffusion of Fe from the substrate into the CIGS layer, preventing in this way the reduction in cell's efficiency [26]. The chemistry of W exhibits many similarities with that of Mo. Despite the fact that the use of alkaline solutions is not recommended in the literature for the dissolution of metallic W either [22], the constructed Pourbaix diagram of W for a concentration as low as the one present in our experiments (Fig. S2c, Supplementary material) showed that dissolution of W is possible through WO_4^{2-} formation, under alkaline conditions, with $\Delta G^0_{20^\circ C} = -200$ kJ/mol W (Fig. S1b, Supplementary material). Regarding Ti, its Pourbaix diagram (Fig. S2e, Supplementary material) indicated the dominance of solid TiO_2 after leaching, for a wide range of pH and electrochemical potentials. When it comes to the CIGS compositional elements, their constructed Pourbaix diagrams showed that mild alkaline conditions (pH $<$ 10) were not expected to considerably dissolve the CIGS, either due to the insolubility of the selenides or formation of insoluble hydroxides. Stronger alkaline conditions though (pH $\geq$ 11) seemed able to affect the CIGS, by dissolving Se to SeO_3^{2-} or SeO_4^{2-}, allowing Cu to form the solid $Cu(OH)_2$ and dissolving Ga to Ga $(OH)_4^-$ (Figs. S2g, i, k, respectively, Supplementary material). It should be clarified that these predictions were based on the use of the individual selenides instead of the whole CIGS compound, as no data for the latter was available in the HSC database or in the literature, meaning that there might be some discrepancies between the predicted and observed leaching behavior of the CIGS. Finally, the construction of the plots for In was compromised, probably due to some missing data in the database. Thus, a simple plot for In only (in the absence of Cu, Ga and Se) was constructed, excluding the species $InOH^+$ (whose presence also caused issues with the construction of the plot). At least for this simple plot, the formation of $In(OH)_3$ dominated the areas of alkaline pH (Fig. S2m, Supplementary material).

To sum up, the following scenario was considered likely to happen when leaching with alkaline solutions: Mo and W were both expected to dissolve and the kinetics of their leaching would determine i) if the liberation and recovery of the CIGS material as particulate matter could take place before the CIGS layer reacted with the leaching agent and ii) if the time required for Mo dissolution would be reasonable, in case of future scale up of the process. According to the basic principles of leaching, mild alkaline solutions were expected to cause lower leaching rates compared to stronger ones. However, the thermodynamic investigation showed that mild alkaline conditions offered the advantage of no losses of the CIGS in the leachate, since the CIGS elements would remain in the solid state, as insoluble selenides and/or hydroxides. On the contrary, the use of strongly alkaline solutions would potentially accelerate the leaching rates, but cause the partial dissolution of the CIGS material, with its compositional elements ending up scattered in different physical states, with an increased need for further separation. Therefore, a range of leaching agent's concentrations had to be investigated, in order to check if the Mo leaching could be both reasonably fast and selective at the same time.

3. Materials and methods

The solar cell samples, experimental procedures and equipment used in this paper are described in this section. All experiments were performed in triplicates. For the cases where analysis of triplicates was not possible, the followed procedure is described in the respective section.

3.1. CIGS solar cell samples

For all experiments, discarded CIGS solar cells manufactured by Midsummer AB, with a stainless-steel substrate and a total surface area of 15.6 $\times$ 15.6 cm^2, were the waste source. Smaller pieces of these cells (4 $\times$ 4 cm^2) were cut and each experimental replicate used one of these pieces as sample (i.e. for one triplicate: replicate#1 used one small piece of cell#1, replicate#2 used one small piece of cell#2 and replicate#3 used one small piece of cell#3, so that cell-to-cell differences are also taken into account). The pristine samples, i.e. before they undergo any treatment at all, will be described by the term "untreated" from now on. Unless otherwise specified, the samples used in this work had previously undergone treatment with US in 0.1 M HNO_3, for removal of all the layers placed above the CIGS, like ZnO, ITO and Ag (a procedure described elsewhere [18]). Therefore, the layers which had remained still attached onto the stainless-steel substrate were the CIGS, Mo, W and Ti-containing layers. These samples are going to be called "unleached" from now on, indicating that they had not undergone the alkaline leaching treatment yet (although they had already been subjected to the US-leaching treatment).

3.2. Chemicals

Ultra-pure water (Milli-Q, Merck) with a resistivity of 18.2 MΩ cm was used for the preparation of all solutions. NaOH pellets (purity>98%, Merck) were used as the leaching agent and HNO_3 65% (Suprapur, Merck) was used in the form of aqueous solutions for the acidification and dilution of the liquid samples which would be subjected to elemental analysis.

3.3. Determination of the total Mo in the untreated solar cell

The average total amount of Mo per untreated solar cell was determined in this paper by leaching three cells at room temperature (20 °C) for up to 32 h in 1 M NaOH, using an orbital shaker at 200 rpm. The geometrical surface area to liquid ratio (A:L) used was 1:3 cm^2/ml (e.g. for the 4 $\times$ 4 cm^2 sample, A = 16 cm^2). The progress of leaching was monitored by analyzing the elemental composition of aliquots taken during the leaching, at pre-determined time points. When the maximum leaching efficiency was achieved, microscopic observation of the undissolved solids took place for confirming that no undissolved Mo had remained. It should be clarified that determination of the total initial concentrations of the rest of the elements present in the solar cell was not considered necessary for this work, therefore, it was not performed.

3.4. Alkaline leaching with 0.1-0.5 M NaOH

Since the kinetics of a direct reaction between low-concentration metallic Mo and NaOH solutions was unknown, a range of different leaching agent concentrations had to be tested. It is known that higher leaching agent concentrations usually increase the reaction rate and efficiency [27], but harsh leaching conditions were to be avoided, due to potential impact on the CIGS compound and sustainability-related reasons. For a compromise between these needs, the concentrations decided for were 0.1, 0.3 and 0.5 M NaOH. Leaching was performed in closed plastic containers at ambient temperature (20 °C), using an

orbital shaker (PSU-10i by Grant-bio) for agitation at 200 rpm. A:L equal to 1:3 cm^2/ml was used. The progress of the reactions was checked by taking aliquots from the leachates during the leaching process, at pre-determined time points, and analyzing their elemental composition. Analysis of the resulting solid fractions followed, when necessary.

Possible formation of H_2 was also checked during the leaching with 0.5 M NaOH, using a H_2 sniffer (Snooper mini, Sewerin), at random time points, in order to obtain some insight into the possible reactions taking place.

3.5. Alkaline leaching using a NaOH solution of constant pH = 11 at 50 °C

After it was shown that Mo leaching can take place within reasonable time when using the previously tested NaOH concentrations, preliminary factorial experiments, described in [28], were conducted for investigating if even lower NaOH concentrations could offer efficient leaching of Mo. These experiments indicated leaching conditions promising for effective and selective leaching of Mo with low NaOH consumption, which were then used in a second set of alkaline leaching experiments for our current work.

More specifically, for this second experimental set, an automatic titration system (905 Titrando, Metrohm) was used for leaching the solar cells with a NaOH solution of constant pH = 11 at 50 °C and A:L = 1:6 cm^2/ml. The titrant was a solution of 1 M NaOH. The leaching process was automatically controlled and all the data (added volume of NaOH, pH and ORP of the leachate) were registered using the Tiamo 2.5 software. The pH of the leachate was measured with a combined pH electrode (6.0260.010, Metrohm), while the ORP of the leachate was measured using a combined Pt ring electrode (6.0451.100, Metrohm). The progress of the leaching was monitored by analyzing the elemental composition of aliquots taken during leaching, at pre-determined time points. For the most promising leaching times, the experiments were repeated, in order to test different liberation methods for the CIGS material after the alkaline leaching, collect the resulting solid fractions and characterize them. More specifically, in one set of experiments, after the desired leaching time was reached, the treated sample was placed in a beaker along with its original leachate and treated in a US bath (model USC-THD/HF, 132 kHz US frequency and 80 W output power, VWR) at the maximum power setting, until all the CIGS material was removed from the substrate or for up to 60 min, if the removal was not complete until that time. In a second experimental set, a different CIGS liberation technique was tested: a new set of replicates was prepared and the process was repeated, but, this time, the CIGS material was removed manually after the leaching, by using a brush (toothbrush of medium softness). Analysis of the solid fractions followed.

3.6. Instrumentation for characterization

The elemental analysis of the leachates was performed using Inductively Coupled Plasma – Optical Emission Spectroscopy (ICP-OES, iCAP PRO Duo, ThermoScientific). External standards were prepared using elemental standard solutions for the elements Cr, Cu, Fe, Ga, In, Mo, Se, Ti, and W (1000 ppm, Inorganic Ventures) and an internal standard of 1 ppm Y was used for all external standards and unknown concentration samples. The matrix of all the external standards was also matched with the matrix of the unknown concentration samples. The latter were also acidified with HNO_3 to a final concentration of about 1 M HNO_3, in order to prevent sample deterioration by possible precipitations. For the same reason, all ICP measurements were made the same day the aliquots were collected.

The morphology of the surface and elemental composition of the untreated solar cells, the leached substrates and recovered solids were studied using Scanning Electron Microscopy coupled with Energy Dispersive X-ray Spectroscopy (SEM-EDS, FEI Quanta 200 FEG-SEM with EDS detector X-Max, Oxford Instruments). The crystalline phases

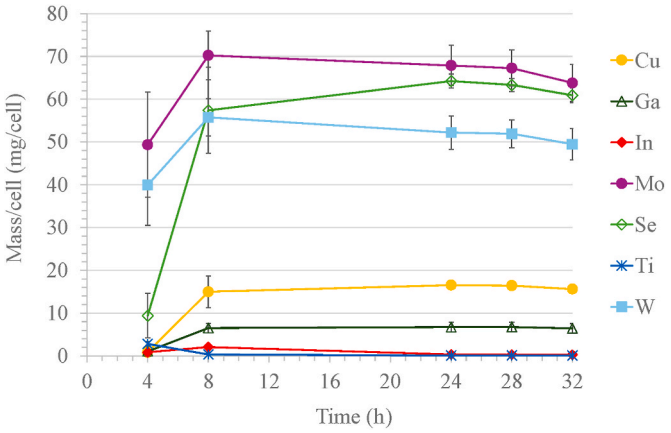

Fig. 1. Determination of the total Mo concentration in CIGS solar cells via leaching with 1M NaOH.

of solid particles were identified with an X-Ray Diffractometer (XRD, D8 Discover, Bruker), using Cu K_α radiation and an air scattering knife placed at 1 mm above the sample level. The EVA software and JCPDS database were used for phase identification. It should be clarified that i) for the analysis of solid material coming from the untreated and unleached cells, the material of interest was first removed manually from the substrate and recovered in particulate form and ii) since the amount of film materials per replicate of the leaching experiments was very small, before any analysis of solid particles took place, the solids coming from the replicates performed under the same conditions were mixed together and this mix was the sample which was analyzed.

4. Results and discussion

In this section, the results of the quantification of total Mo content in the untreated samples are presented first, followed by the results of the elemental composition of the unleached CIGS compound. Finally, the results of the leaching experiments performed with NaOH concentrations of 0.1-0.5 M and a NaOH solution of pH = 11 at 50 °C are presented. It should be clarified that although Fe and Cr were also measured in all aliquots, these elements were omitted from all the leaching plots which follow, since their concentrations were always close to or below their detection limits. It should also be noted that all presented errors correspond to 1σ standard deviation for the triplicate mean values.

4.1. Determination of the total Mo in the untreated solar cell

A complete leaching of Mo was achieved after 8 h of leaching the untreated cells with 1 M NaOH, showing 70.3 ± 5.7 mg Mo/cell (Fig. 1). The completion of its dissolution was confirmed via EDS analysis, which showed the absence of Mo from the digested substrates and suspended solids (Section S2, Supplementary material). Although W was not an element of focus, it is interesting to note that the specific conditions led to its complete dissolution too, showing 55.8 ± 4.4 mg W/cell.

4.2. Determination of the composition of the unleached CIGS

The SEM-EDS analysis of the unleached CIGS material revealed the presence of mainly Cu, Ga, In and Se in it, as expected. A peak assigned to S was also detected. Since it was not clear how much of the detected S had to be assigned to the CIGS layer (some S is usually added in it during manufacturing [29]) and how much was coming from the S present in the sticky pad used for the analysis, the composition of the CIGS was calculated in two ways, considering the following two extreme scenarios: first, the presence of S was completely ignored, as if it was only due to the sticky pad. Second, the whole amount of S detected was

Table 1
Average wt% and atomic% composition of the unleached CIGS material (average of 6 measured areas of the sample).

	With S				Without S			
	Wt%		Atomic%		Wt%		Atomic%	
	Average	σ	Average	σ	Average	σ	Average	σ
Cu	15.8	0.2	20.1	0.4	16.1	0.2	21.1	0.3
Ga	5.6	0.2	6.5	0.3	5.7	0.2	6.8	0.2
In	29.8	0.5	20.9	0.3	30.3	0.7	21.9	0.5
Se	46.8	0.8	47.8	1.1	47.8	0.5	50.2	0.5
S	1.9	0.6	4.7	1.4	-	-	-	-

assigned to the CIGS material. The difference between the two extreme values was very small (Table 1).

4.3. Alkaline leaching using 0.1-0.5 M NaOH

When leaching with 0.1, 0.3 and 0.5 M NaOH for up to 24 h (Fig. 2), complete leaching of both Mo and W was possible, for all the NaOH concentrations tested. The main difference between the leaching plots was the dissolution rate: the higher the NaOH concentration, the faster the dissolution curves reached a plateau. More specifically, when leaching with 0.1 M NaOH, Mo and W needed about 24 h in order to dissolve completely. However, when 0.3 and 0.5 M NaOH were used, the leaching reaction of both elements was completed after about 8 h. It is worth mentioning here that evolution of H_2 gas was detected, indicating that either i) the alkaline leaching of Mo proceeded through reaction (1) (and similar could be the case for W too, as explained in Section 2), or ii) the $MoSe_2$ layer acted as a catalyst for H_2 evolution or iii) more than one reactions forming H_2 were taking place simultaneously. The leaching rate of Cu, Ga, In and Se was also affected by the NaOH concentration: for the same treatment time, higher amounts of these elements were leached into the solutions of higher NaOH concentration. Finally, the concentration of Ti in the leachate was negligible in all cases.

Based on these results, there was no NaOH concentration which was clearly preferable over the others for selective dissolution of Mo. The conditions which looked promising for further investigation though were the following: a) leaching with 0.1 M NaOH for 8 h or 24 h, b) leaching with 0.3 M NaOH for 8 h and c) leaching with 0.5 M NaOH for 8 h. More specifically, leaching with 0.1 M NaOH offered the advantage of lower consumption of chemicals and less contamination of the leachate with Na ions, as well as selective leaching of Mo and W over Cu, Ga and In. However, a considerable amount of Se was leached during the about 24 h required for the complete leaching of Mo and W (Fig. 2a). Moreover, XRD analysis of the suspended solids (Fig. 3) showed that although their main crystalline compound was still the CIGS, of the same crystalline structure as the untreated CIGS, some $In(OH)_3$ had also formed. This heterogeneous solid fraction can be challenging to separate later. Reduction of the leaching time to 8 h could decrease the reaction rate of the CIGS compositional elements, however, the leaching efficiency of Mo and W would be only 70-75 wt% in this case (Fig. 2a). That could cause difficulties in the subsequent liberation of the CIGS material from the substrate, as well as possible contamination of the recovered CIGS material with still intact Mo and W. If the highest NaOH concentration tested (i.e. 0.5 M) is used instead, the higher amount of chemicals is certainly a disadvantage. However, the leaching of Mo and W in this case was completed within only 8 h (Fig. 2c), considerably faster compared to the case of 0.1 M NaOH. Although the losses of Cu, Ga, In and Se in the leachate at 8 h were higher as well when leaching with 0.5 M NaOH, the CIGS compound still did not dissolve completely and the suspended solids consisted, again, mainly of CIGS, as well as an amount of $In(OH)_3$ (Fig. 3). Finally, leaching with 0.3 M NaOH resulted in a situation between the one for 0.1 M NaOH and the one for 0.5 M NaOH: Mo and W were leached completely in about 8 h, however, a considerable amount of Se was also leached within this time, along with some In, Cu and Ga (Fig. 2b). Due to the profound similarities with the previously discussed cases, XRD analysis of the resulting suspended solids did not take place.

To sum up, leaching with 0.1-0.5 M NaOH at 20 °C proved successful

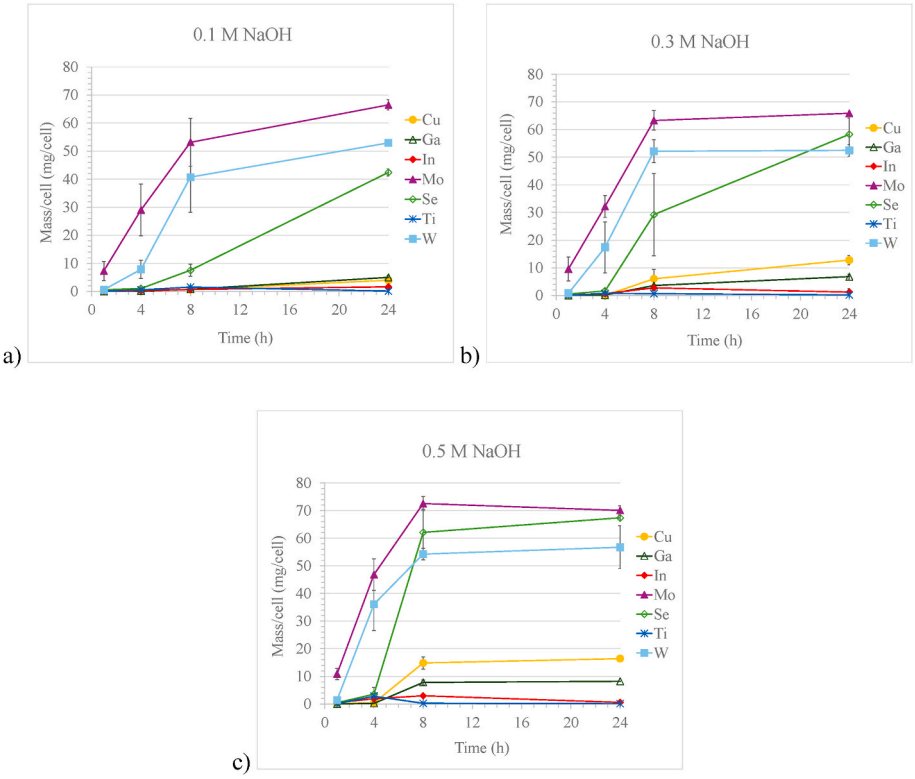

Fig. 2. Leaching yields of elements vs time for leaching with a) 0.1 M, b) 0.3 M and c) 0.5 M NaOH at 20 °C.

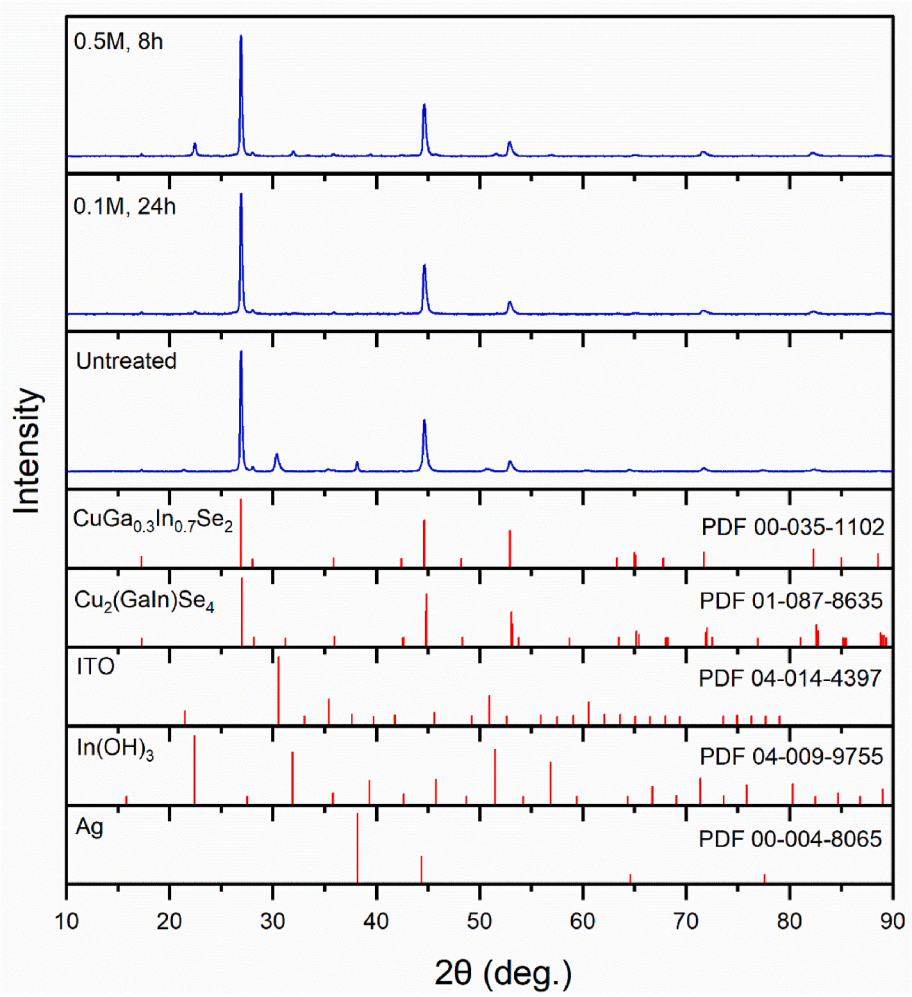

Fig. 3. XRD analysis of the untreated CIGS layer and the suspended solids collected after leaching with 0.1 M and 0.5 M NaOH for 24 h and 8 h, respectively. (The compounds which were present in the untreated cell, but removed before the alkaline leaching experiments took place, are not identified in detail, except for the strongest peaks, belonging to Ag and ITO.)

for complete dissolution of metallic Mo and W, as predicted by thermodynamics. However, the time required for the completion of the reaction depended greatly on the NaOH concentration. At the same time, complete Mo and W leaching was always accompanied by considerable leaching of Se too, again, in good agreement with the thermodynamic predictions. Regarding Cu, Ga and In, their leaching rates increased with the NaOH concentration and an amount of In also precipitated under all tested conditions as $In(OH)_3$. The fate of these metals was not in complete agreement with the thermodynamic predictions, as they ended up scattered in various phases. That could be a result of slow reaction kinetics or just poor thermodynamic predictions, due to the lack of the CIGS compound in the HSC database. In any case, these experiments suggested that moderate concentrations of NaOH are effective for complete leaching of metallic Mo, but improvements in the process were necessary, in order to achieve reasonably fast Mo leaching without reaction of the CIGS compound.

4.4. Alkaline leaching using a NaOH solution of constant pH = 11 at 50 °C

When the leaching conditions of constant pH = 11 (adjusted by continuous addition of NaOH) and T = 50 °C were applied, the leaching

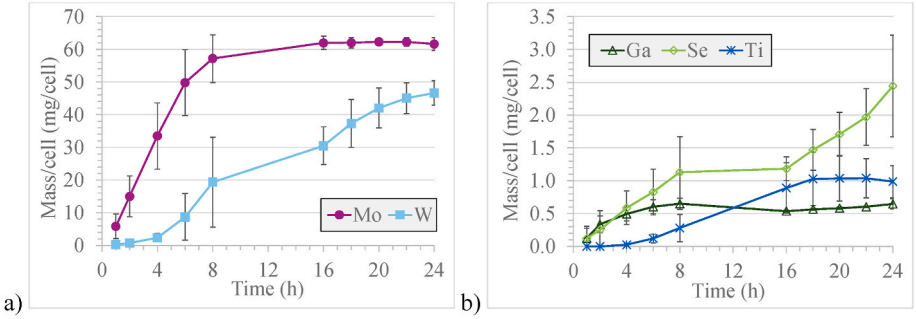

Fig. 4. Leaching yields of the a) major and b) minor elements of the leachate when leaching with a NaOH solution of pH = 11 at 50 °C.

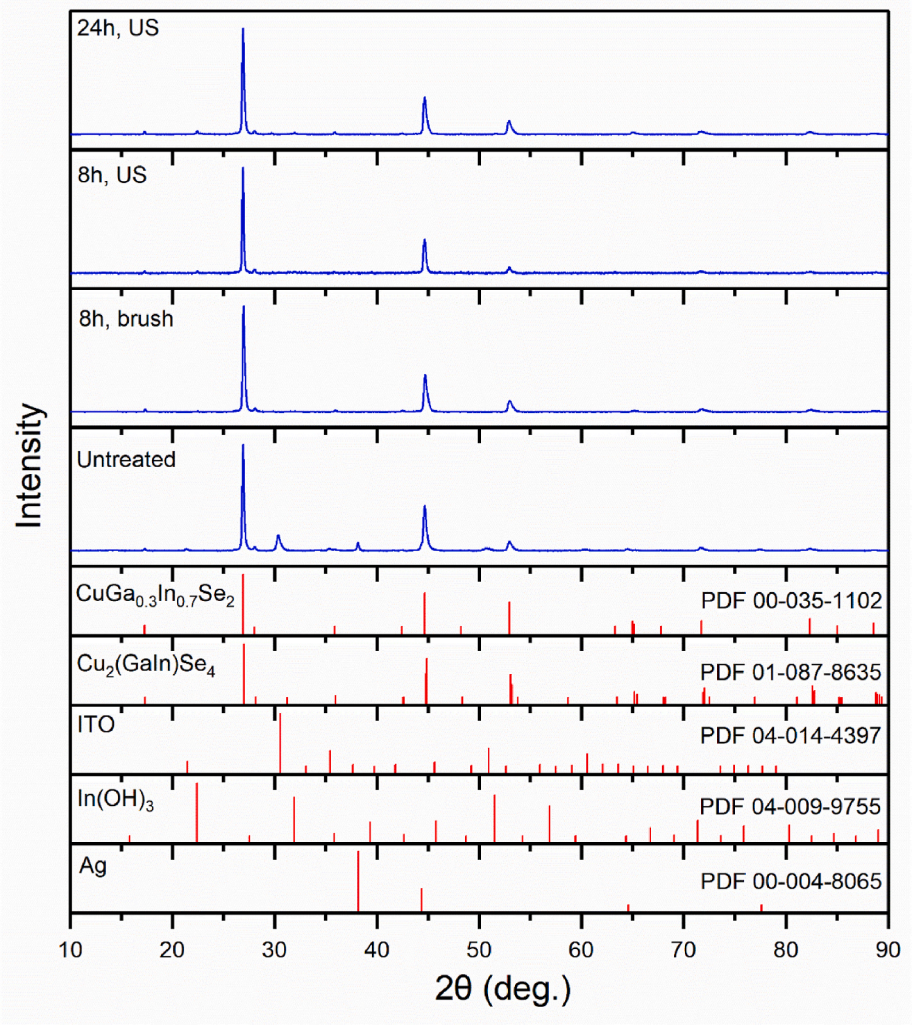

Fig. 5. XRD analysis of the untreated CIGS layer and the suspended solids collected after leaching with a solution of pH = 11 at 50 °C for 8 h and 24 h and subsequent ultrasonication or brushing for the removal of the CIGS material. (The compounds which were present in the untreated cell, but removed before the alkaline leaching experiments took place, are not identified in detail, except for the strongest peaks, belonging to Ag and ITO.)

yield of Mo reached a plateau after 16 h (Fig. 4), but W needed at least 24 h until its yield plateaued. It is worth noting that none of the two elements dissolved completely, as the maximum leaching yields reached 89 wt% and 84 wt% for Mo and W, respectively. Regarding the rest of the elements, only some small amounts of Se, Ga and Ti (no Cu or In) were detected in the leachate, even at 24 h. More specifically, the leaching of Se increased with time, however, it remained well below 3.5 mg/cell, meaning that less than 5 wt% of the maximum leachable Se (based on the maximum leachable amounts of Fig. 1) was lost in the leachate even at prolonged treatment times. The leaching of Ga and Ti reached a plateau after 6 and 18 h, respectively, resulting in the leaching of less than 10 wt% and 33 wt% of their maximum leachable amounts, respectively ([8] and Fig. 1). It is worth noting that the ORP value measured during these experiments remained between −70 and −30 mV (Fig. S4a, Supplementary material), confirming the absence of strong oxidizing or reducing conditions during the process. In a nutshell, these new leaching conditions achieved more selective leaching of Mo over the CIGS, compared to the higher NaOH concentrations investigated in the previous section, as even prolonged residence times of 24 h did not affect now significantly the composition of the CIGS material and, at the same time, achieved considerable leaching yields for Mo and W.

Based on these results, leaching for 24 h was considered promising for further optimization and, thus, the leached sample was subjected to ultrasonication, for liberation of the remaining CIGS material from the leached substrate. The latter step exhibited vastly different times required for the CIGS liberation among the replicates; from 15 min to longer than 60 min (Fig. S5a, Supplementary material). The recovered solids had the same crystalline structure as the untreated CIGS (Fig. 5). A small peak of $In(OH)_3$ was also detected (Fig. 5), likely formed due to the action of US, as explained later. The other treatment time which looked promising for further investigation was the 8 h, as i) a considerable leaching yield of Mo (~82 wt%), ii) a very low leaching yield of Se (no more than 2 wt%), and iii) only traces of Ti contaminating the Mo-bearing leachate were observed for this time. Therefore, after the alkaline leaching was performed for 8 h, the possibility of liberating the CIGS using US was investigated again. This strategy proved unsuccessful, since most of the CIGS remained attached on the substrate of the cell even after 60 min in US (Fig. S5b, Supplementary material), most likely due to the reduced leaching yields of Mo and W. The collected solids consisted, again, mainly of CIGS as well as some $In(OH)_3$ (Fig. 5).

When brushing was tested as an alternative method to US for CIGS liberation after 8 h of leaching, the former proved successful, with the required brushing times ranging from a few seconds up to 25 min (Fig. S5c, Supplementary material). This treatment combination was considered as the most promising and further detailed analysis of the solid fractions followed. More specifically, a considerable part of the surface of the brushed substrates had an iridescent color, indicating the absence of the dark-colored CIGS (Fig. S5c, Supplementary material).

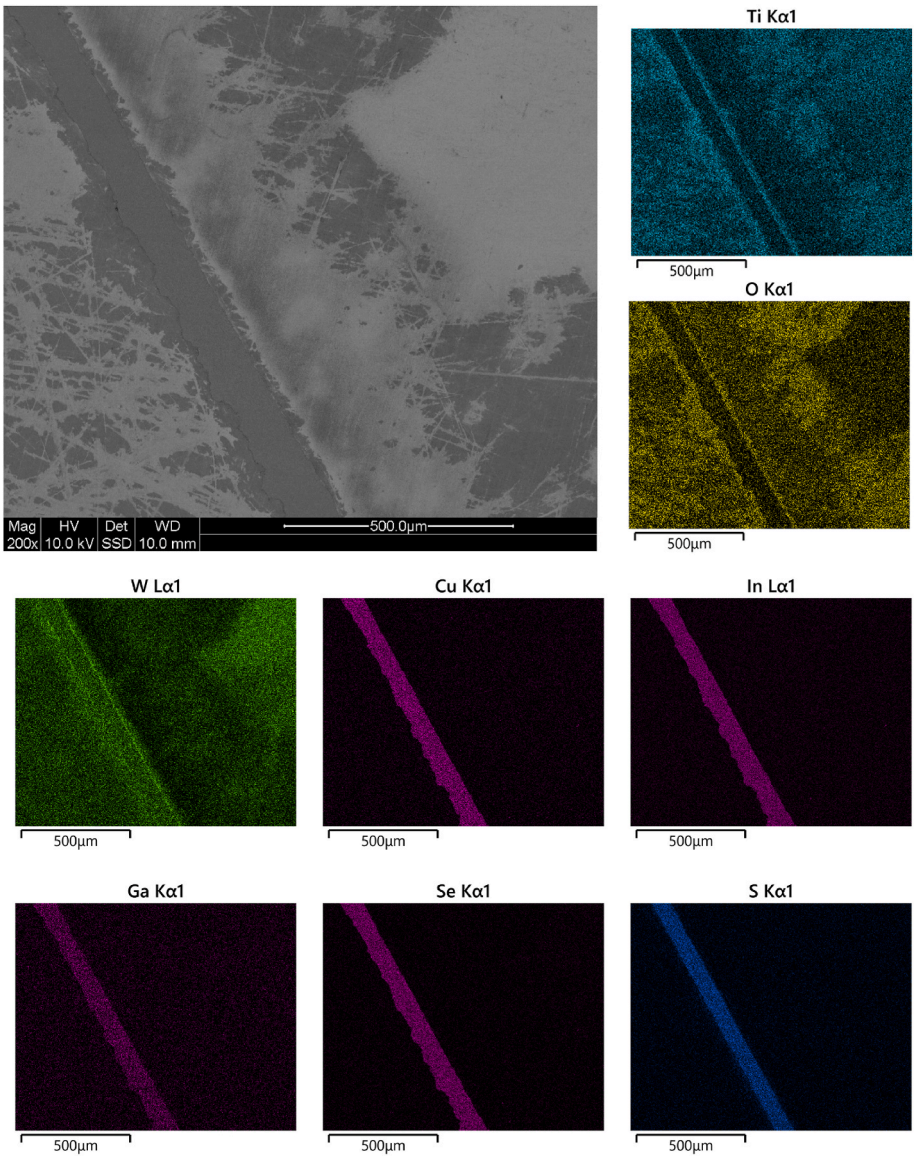

Fig. 6. SEM-EDS mapping of an area of the brushed substrate. Before brushing, the solar cell had been subjected to leaching with a NaOH solution of pH = 11 at 50 °C for 8 h.

SEM-EDS morphological analysis and elemental mapping of the treated substrate (Fig. 6) confirmed the successful removal of the CIGS layer from the bulk surface and showed that only some traces of CIGS were left, trapped in the areas where the Ag lines used to be before their removal. The case was exactly the same for Mo, although the very small peak detected could be assigned to S as well, as the strongest EDS peaks of these two elements overlap (thus, only the map for S was decided to be presented in Fig. 6). The presence of both elements was possible, as, on one hand, S is used sometimes in the synthesis of the CIGS layer and, on the other hand, some traces of undissolved Mo may have remained trapped along with the CIGS after the alkaline leaching. The only elements detected on the bulk surface of the substrate were W, Ti and O. Notably, Ti and O were detected at the same areas, implying the presence of an oxide, most likely TiO_2, according to the thermodynamic predictions (Fig. S2, Supplementary material), therefore, the phase will be called like that from now on. The analysis of the liberated particulate solids showed that they consisted of CIGS of the same crystalline structure as the untreated CIGS and, notably, no other crystalline compounds were detected (Fig. 5). This suggested that the $In(OH)_3$ present in the solid fraction in the previous cases (i.e. of treatment with US after the alkaline leaching) had probably formed due to the action of the US. That gives extra credit to the removal of the CIGS by brushing, since this method resulted in a more homogeneous solid fraction. SEM-EDS mapping of the recovered particles confirmed the dominance of the CIGS compound, but it also detected a very small amount TiO_2 (Fig. 7). The latter should have come from the scratching of the TiO_2 layer by the brush, as the SEM picture of the substrate suggests (Fig. 6). A small amount of Mo was also detected by EDS in the solid particles; something expected, due to the incomplete leaching of Mo. Again, some S may have been present too (as the strongest peaks of Mo and S coincide), if added to the CIGS material intentionally or as a background signal coming from the sticky pad used for the SEM-EDS analysis. The total elemental composition of the recovered solid particles based on the EDS analysis is presented in Table 2. The elemental composition of only the recovered CIGS compound is also presented in the same table. By comparing these compositions with the ones of the unleached CIGS (Table 1), it is confirmed that the composition of the CIGS remained practically unaffected throughout the whole process. It is also worth noting that W was absent from the recovered solids (i.e. all solid W had remained on the substrate) and, also, the total concentration of Ti in the recovered

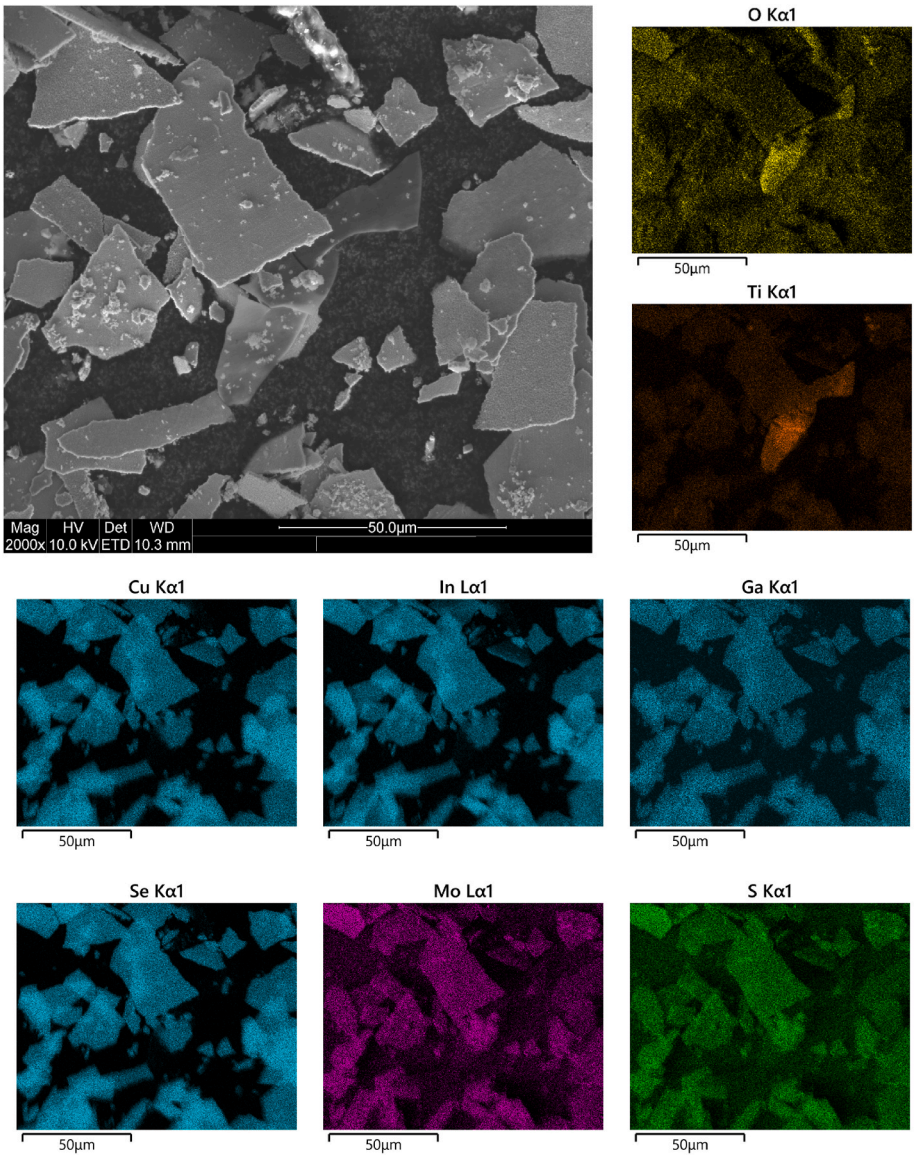

Fig. 7. SEM-EDS mapping of the solids recovered after brushing a cell which had previously been leached with a NaOH solution of pH = 11 at 50 °C for 8 h.

fraction was below the detection limit of the EDS. Based on the results of Table 2, the CIGS content in the recovered solids was about 95 wt%.

To sum up, when leaching with a NaOH solution of constant pH = 11 at 50 °C, most of the Mo (~82 wt%) dissolved after 8 h, along with some W. The CIGS material was then effectively recovered by brushing. The analyses showed that the CIGS remained practically unaffected in terms of chemical composition and crystalline structure throughout the whole recycling process. The recovered CIGS had a purity of about 95 wt%.

4.5. Total assessment of the different treatment methods

In total, the experiments performed in this paper showed the ability of metallic Mo to dissolve in NaOH solutions, under all tested conditions. The concentration of NaOH and temperature of leaching affected the reaction rates and yields of the majority of the elements present in the CIGS solar cell, with higher concentrations and temperatures favoring faster rates and higher yields, as expected according to fundamental chemistry principles. As a result, although the latter conditions increased the leaching of Mo and W, this was also the case for the leaching of the valuable CIGS elements, with a considerable amount of Se ending up in the leachate, some In precipitating as $In(OH)_3$ and some Ga being leached out of the CIGS material too. This undesirable behavior had partly been predicted by the thermodynamic simulations, although it seems that kinetics-related reasons did not allow the predicted reactions to be completed. Low NaOH concentrations of pH = 11 at 50 °C resulted in even slower kinetics of the reactions of the CIGS with the alkaline leaching agent, enabling the selective leaching of Mo, as they were still efficient enough for Mo dissolution. It should be noted here that the A:L is also expected to have affected the leaching efficiency. However, when A:L is in the range of 1:3 - 1:6 cm^2/ml, its effect on the leaching efficiency of Mo is weaker than that of NaOH concentration and temperature [28]. Further research would be required in order to determine the exact mechanism behind the Mo dissolution in the studied system, as the system's complexity is high, due to the presence of many different materials. Finally, after leaching, the liberation of the unleached CIGS material from the stainless-steel substrate proved more efficient when performed by brushing, compared to US. After alkaline leaching for 8 h and subsequent liberation of the CIGS by brushing, the composition and crystal structure of the recovered CIGS remained practically unaffected throughout all recycling steps. The purity of the recovered CIGS was about 95 wt%, with contamination coming mainly from some unleached Mo, as well as traces of the TiO_2 layer scratched by

Table 2
EDS analysis of the solids brushed out of a cell leached at pH = 11 and 50 °C for 8 h (average of 3 measured areas of x200 magnification).

	Total solids							
	With S				Without S			
	Aver. wt%	σ	Aver. atomic%	σ	Aver. wt%	σ	Aver. atomic%	σ
Cu	15.2	0.2	19.3	0.3	14.9	0.2	19.4	0.2
Ga	5.2	0.1	6.0	0.1	5.0	0.1	6.0	0.1
In	26.0	0.6	18.3	0.4	25.8	0.6	18.6	0.5
Mo	3.2	0.1	2.7	0.1	6.5	0.5	5.7	0.5
S	1.3	0.2	3.4	0.6	-	-	-	-
Se	48.8	0.6	49.9	0.6	47.5	0.8	49.9	0.6
Ti	0.3	0.0	0.5	0.0	0.3	0.0	0.5	0.0
	CIGS only							
	With S				Without S			
	Aver. wt%	σ	Aver. atomic%	σ	Aver. wt%	σ	Aver. atomic%	σ
Cu	15.6	0.2	19.5	0.3	16.0	0.2	20.7	0.1
Ga	5.3	0.1	6.0	0.1	5.4	0.1	6.4	0.1
In	26.4	0.6	18.2	0.4	26.9	0.7	19.2	0.6
S	2.3	0.2	5.7	0.6	-	-	-	-
Se	50.4	0.6	50.6	0.7	51.7	0.5	53.7	0.4

the brush. The recovered CIGS can now be purified further (different methods can be tested for that), in order to finally achieve the required purity of 5N for use in solar cells [15].

5. Conclusions

In this paper, a method for highly efficient selective recovery of the valuable CIGS layer from solar cells was demonstrated. The CIGS layer was recovered practically intact after leaching the solar cell with a NaOH solution of constant pH = 11 at 50 °C for 8 h, which dissolved the Mo-containing layers placed underneath the CIGS, and subsequently brushing the remaining CIGS off the substrate. The purity of the recovered CIGS was about 95 wt%. The experiments also showed that a range of NaOH concentrations can dissolve the metallic Mo layer even at ambient temperature, at least for the low concentrations of Mo used in solar cells. However, the lower the NaOH concentration, the less the CIGS material gets affected and, thus, the more selective its recovery is. In total, the particular paper proved that mild alkaline leaching conditions can be used, in order to selectively recover the valuable CIGS material, simultaneously recover the important for the modern economy Mo and avoid excessive use of chemicals or energy, compared to other stages which have been suggested so far for the recycling of CIGS-containing waste. Therefore, the presented method is very promising for the recovery of CIGS from waste solar cells and even further optimization (e.g. more detailed exploration of the effect of the common leaching parameters as well as the redox potential of the solution on the leachability of the elements, purification of the already recovered solid, etc) would be worth investigating.

CRediT authorship contribution statement

Ioanna Teknetzi: Writing – original draft, Visualization, Validation, Methodology, Investigation, Formal analysis, Conceptualization. **Hai Co Nguyen:** Investigation. **Stellan Holgersson:** Writing – review & editing, Supervision. **Burçak Ebin:** Writing – review & editing, Supervision, Resources, Project administration, Funding acquisition.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.solmat.2026.114237.

Data availability

Data will be made available on request.

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