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OPTIMIZATION OF SOXHLET EXTRACTION PARAMETERS FOR GEL CONTENT DETERMINATION OF CO-EXTRUDED EPE

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ABSTRACT: With the transition to TOPCon technology raising several challenges regarding reliability and long-term stability of PV modules, tailored encapsulant solutions like EPE (EVA+POE+EVA) are more commonly used in PV module production due to their advantageous properties. The optimization and adaptation of precise quality control is hereby most important. While the measurement of Gel Content (GC) with DMA, DSC and FTIR are relative methods, Soxhlet offers a more accurate and reliable quantitative alternative. This study focuses on optimizing Soxhlet extraction parameters for GC measurements of co-extruded EPE encapsulants. The influence of various extraction cycle times as well as number of cycles and resting time in cooling solvent on GC values is investigated. Furthermore a sweep of extraction time from 8-24 h is performed for two different EPE and POE encapsulants to identify the point at which the change in GC saturates. At this point any further increase in extraction time will not lead to significant changes in GC values. With EPE this point of saturation was found at 22-24 h while POE exhibits this saturation behavior at 20-26 h minimum extraction time highly depending on the specific material.

Keywords: Soxhlet extraction, degree of cross-linking, gel content, co-extruded EPE, quality control

1 INTRODUCTION

With the progressive transition from PERC (Passivated Emitter and Rear Cell) to TOPCon (Tunnel Oxide Passivated Contact) several challenges regarding reliability, long-term stability and failure modes of TOPCon came to light [1]. TOPCon is much more susceptible to high humidity, contamination and PID (potential-induced degradation) than PERC as *C. Sen et al.* have shown [1]. According to forecasts from the International Technology Roadmap for Photovoltaic (ITRPV 2025), the market share of TOPCon technology is expected to rise to up to almost 70 % by 2029, thereby replacing PERC as the dominant cell technology [2]. The development of tailored encapsulants and corresponding BOMs is therefore most important [1].

S.K. Chunduri and *M. Schmela* also reached a similar conclusion in the TaiyangNews Market Survey 2022-2023 suggesting the usage of co-extruded EPE consisting of a multilayer system of EVA+POE+EVA [3]. This material combines the advantageous mechanical properties of EVA (ethylene-vinyl acetate) with the low water vapor permeability of POE (polyolefin elastomer) and is priced between these two [4–6].

Due to this combination of properties, the market share of co-extruded EPE encapsulants is expected to rise to up to about 45 % by 2035 according to forecasts from the ITRPV 2025, thus replacing EVA as the dominant encapsulant material [2]. Therefore EPE will be the alternative encapsulant for bifacial products in GG (glass-glass) combinations [2].

For quality control of laminated PV modules DMA (Dynamic Mechanical Analysis), DSC (Differential Scanning Calorimetry) and FTIR (Fourier Transform Infrared Spectroscopy) are commonly used methods for determining GC (Gel Content) of cross-linked encapsulant material [7]. In comparison to these methods, Soxhlet extraction is based on washing out non-cross-linked monomers using a suitable solvent [8]. On the one hand this process is significantly more time-consuming, on the other hand it is a quantitative method and more precise and reliable [8]. In case of EVA a Soxhlet extraction time of up to 16 hours can be expected while POE needs up to 24 hours highly depending on the specific material [8]. Since there is no recommendation for determining the GC of co-extruded encapsulants, the optimization of Soxhlet extraction parameters for GC determination is essential to reliably assess the quality of laminated PV modules using these encapsulant solutions.

The goal of this study is to find suitable process parameters for determining the GC using Soxhlet extraction for co-extruded EPE. For this, the total extraction time as well as cycle time and number of extraction cycles are varied in case of POE and EPE with a high degree of cross-linking. The extraction time is varied from 8-24 h in 1 h increments. The recommended extraction time is determined at which the change in the measured GC saturates. At this point a complete extraction is ensured and any further increase in extraction time no longer has a significant effect on the measured GC.

2 MATERIALS AND METHODS

To compare the GC behavior of POE and EPE using Soxhlet extraction, the materials *POE-A* and *EPE-A* of a Turkish solar encapsulant film manufacturer were used, as well as *POE-B* and *EPE-C* from two different Chinese solar encapsulant film manufacturers. For producing samples for the Soxhlet extraction with a high degree of cross-linking, the POE and EPE materials were laminated according to their longest recommended datasheet processes using a *Ypsator* PV-module laminator from *Robert Bürkle GmbH.* The used materials are listed in Table 1 along with their respective thicknesses.

To be able to extract the double layer of cross-linked encapsulant from the mini – GBS (glass-backsheet) laminates (280x250 mm) for Soxhlet measurements, Teflon sheets are added in between the different layers as seen in Figure 1.

Table 1: List of used materials and their specifications.

Layer	Material	Thickness [mm]
POE-A	POE	0.740
POE-B	POE	0.500
EPE-A	EPE	0.650
EPE-C	EPE	0.550
Glass	Solar glass	3.000
Backsheet	PET/Primer	0.218
Teflon sheet	Polytetrafluoroethylene (PTFE)	0.080

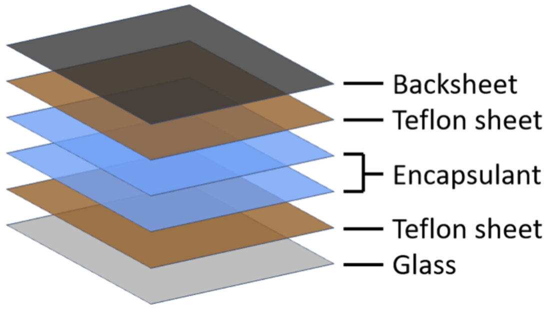

Figure 1: Module lay-up without cells for Soxhlet sample preparation.

In addition to the gel content determination by Soxhlet extraction, the performance of mini-modules with *EPE-A* and *POE-A* encapsulants was evaluated after reliability tests. *Damp Heat* (DH), *Thermal Cycling* (TC) and *Ultraviolet* (UV) aging tests were conducted in accordance with *IEC 61215-2* using the parameters stated in Table 2.

Table 2: Reliability test parameters for mini-modules with *EPE-A* and *POE-A*.

Test	Parameters
***Damp Heat* (DH)**	1000 h at 85 °C / 85 % RH
***Thermal Cycling* (TC)**	200 cycles from -40 °C to 85 °C
DH200 + UV60	200 h DH + 60 kWh/m^2 UV

2.1 Soxhlet Extraction

For Gel Content determination via Soxhlet extraction a *Behrotest* Soxhlet extraction unit was used in accordance with *IEC 62788-1-6*. Each sample was measured 3 separate times. Each datapoint of the results represents the mean value of these separate measurements. Unless stated otherwise a cycle time of 3.5 min is used resulting in about 17 extraction cycles per hour implemented by an integrated siphon-system to continuously cycle fresh solvent. The Gel Content was calculated by the following formular [9,10]:

$$GC\ [\%] = \left(\frac{M_2 - M_0}{M_1 - M_0}\right) * 100,\ M_2 \leq M_1 \qquad [9,10]$$

Each sample was weighed in to 1±0.05 g and placed in a cylindrical stainless steel mesh tube. M_0 represents the pre-determined weight of the tube with M_2 representing the total weight of the sample including the tube before the extraction process. M_1 represents the total weight of sample and tube after extraction and subsequent drying in a vacuum oven at 100 °C for 7 h.

3 RESULTS AND DISCUSSION

3.1 Influence of Soxhlet cycle time and number of cycles

The influence of Soxhlet cycle time on the measured GC was tested by varying the cycle time from 2-5 min (30-12 cycles/hour) with intervals of 1.5 min. The results are shown in Table 3 for EPE-C after a constant extraction time of 6 h therefore effectively varying the total amount of extraction cycles.

Decreasing the cycle time from 5 to 2 min leads to a change of only -0.43 % in measured GC. The influence of varying the Soxhlet cycle time on the measured GC with a constant extraction time is therefore not significant.

Table 3: GC values of EPE-C after 6 h extraction with different cycle times.

Cycle time [min]	No. of extraction cycles [ ]	Gel Content [%]
2	180	91.57
3.5	~100	91.67
5	72	92.00

To further test the influence on the measured GC a Soxhlet extraction was carried out with a constant number of 100 cycles while varying the cycle time from 2-5 min with intervals of 1.5 min therefore varying the total extraction time from 200-500 min as shown in Table 4.

Decreasing the number of cycles from 500 to 350 and to 200 leads to significant changes of +1,23 % and +2,68 % in measured GC respectively. Since the number of cycles was held constant, this change in GC can be explained by the total extraction time. A longer extraction time necessarily leads to more non-cross-linked monomers and/or low molecular weight polymer chains being washed out therefore resulting in a decrease in measured GC.

Table 4: GC values of EPE-C after 100 extraction cycles with different cycle times.

Cycle time [min]	Total extraction time [min]	Gel Content [%]
2	200	94.42
3.5	350	92.97
5	500	91.74

3.2 Influence of resting time in cooling Soxhlet solvent

To investigate the influence of additional resting time in the cooling solvent after a finished Soxhlet extraction a 6 h extraction of *EPE-C* was performed. After the finished extraction the samples were taken out of the Soxhlet apparatus and dried in a vacuum oven after different resting times. The first sample was taken out after 0.75 h (45 min) while the second sample rested in the solvent for 3 h (180 min). The third sample was taken out on the following day after a total amount of 18 h (1080 min) of resting time in the solvent. In Table 5 the measured GC values are shown. Due to the heating plates of the *Behrotest* Soxhlet apparatus needing to cool down, the usual amount of resting time is about 20-30 min before being able to take out samples.

After 45 min a GC of 91.78 % was measured. After a resting time of +3 h a decrease of 1.39 % could be measured compared to the initial GC value of 91.78 %.

When comparing the measured GC values after +3 h and +18 h of resting time this initial decrease of GC is no longer visible. In this case no significant change on GC can be measured.

This behavior can be explained by the remaining temperature of the solvent. Until it cools down non-cross-linked monomers and/or low molecular weight polymer chains are still being extracted from the sample since the diffusion coefficient is still high enough for effectively washing out compounds. With a decrease in temperature this diffusion process slows down heavily resulting in no more significant changes in GC after +18 h resting time at room temperature.

Table 5: GC values of EPE-C after additional resting time in cooling solvent after a finished 6 h extraction.

Resting time [h]	Gel Content [%]
+ 0.75	91.78
+ 3	90.39
+ 18	90.47

Lust et. al. encountered a similar effect in their study [11]. In their case a 5 h extraction was conducted with additional heating/cooling steps with 15 h of overnight resting time resulting in a significant decrease of 8.1 % compared to a 5 h extraction without resting time [11]. This highlights the importance of a set amount of time until taking out the samples. Otherwise, the results can become highly distorted and will no longer be reproducible.

3.3 Variation of Soxhlet extraction time

To determine suitable process parameters for GC measurement of co-extruded EPE using Soxhlet the extraction time was varied from 8-24 h in 1 h increments. To ensure a complete extraction another set of samples was measured after 32 h extraction. An EVA encapsulation film of a Chinese solar encapsulant film manufacturer is used as reference and is therefore listed as *EVA ref* in the following diagrams. The results of this extraction time sweep from 8-24 h can be seen in Figure 2 for both *EPE-A* and *EPE-C*. The material *EPE-A* was laminated at 155°C for 20 min while *EPE-C* was laminated at 150°C for 18 min. These lamination parameters represent the longest recommended datasheet processes.

Both EPE encapsulants exhibit considerably higher GC values than the *EVA ref* material. At the lowest extraction time of 8 h *EPE-A* and *EPE-C* reach GC values of 93.8 % and 92.51 % respectively while *EVA ref* reaches 86.51 %. At the highest extraction time of 24 h 90.57 % and 86.03 % are reached. Over the period of the 8-24 h extraction time sweep *EPE-A* therefore shows a decrease of 3.23 % while *EPE-C* shows a decrease of 6.48 % which is twice as high. After 32 h extraction time a decrease of 0.15 % in case of *EPE-A* and 0.3 % in case of *EPE-C* can be seen in comparison with the GC values after 24 h. Since 8 h more extraction time leads to an insignificant decrease in GC for both EPE encapsulants a complete extraction can be assured.

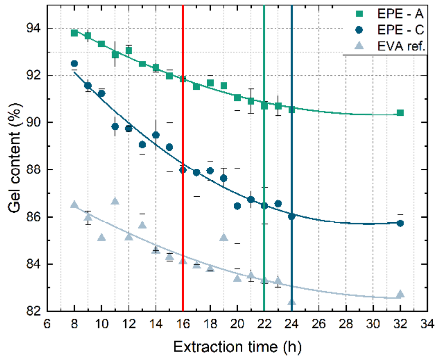

Figure 2: Measured gel content of EPE encapsulants as function of extraction time (3 samples per data point) with fitted curves and calculated minimum extraction time (green & blue line) in comparison with recommended extraction time for POE [12] (red line).

The changes in GC values of both EPE encapsulants are shown in Figure 3. The measured data exhibits volatile jumps in GC values above and below the 0 %-line due to unavoidable measurement inaccuracies. This is exceptionally true for *EPE-C* which shows several jumps above the line at high extraction times as seen in Figure 3. To be able to graphically determine the extraction time where the GC saturates, the curves of both EPE encapsulants were individually fitted as shown in **Error! Reference source not found.** in case of *EPE-A*.

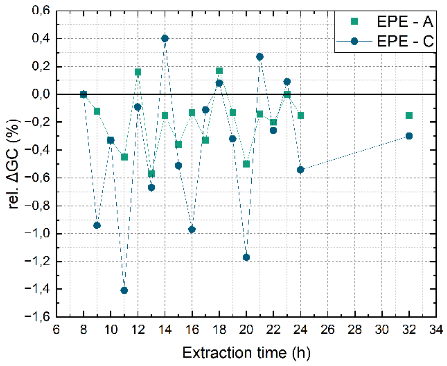

Figure 3: Relative change in measured gel content of co-extruded EPE encapsulants as function of extraction time (3 samples per data point).

The data of these individually fitted curves was then used to calculate the change in GC values of both EPE encapsulants as seen in Figure 5. To determine the gel content at the saturation point the change of GC was analyzed at the intersection with the 0 %-line.

In case of *EPE-A* this saturation is reached at 31 h of calculated extraction time with a GC value of 90.34±0.52 % which is well above the recommended extraction time for POE of 16 h [12]. Taking the calculated standard deviation of 0.52 % into account the minimum extraction time to reach the measured GC value of 90.34 % is 22 h.

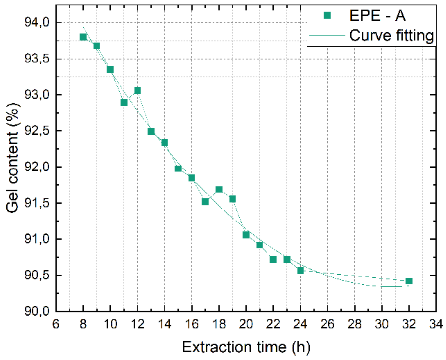

Figure 4: Measured gel content of co-extruded *EPE-A* as function of extraction time (3 samples per data point) with fitted curve.

In case of *EPE-C* the point of saturation is reached at 30 h of calculated extraction time with a GC value of 85.70±0.52 % as seen in Figure 5. With the standard deviation of 0.52 % the GC value of 85.70 % can already be reached at 24 h of Soxhlet extraction. This coincides with the recommended 24 h for certain POE encapsulant materials [8]. It should be noted that this might not be the case for badly cross-linked material. Since the recommended extraction time for EVA encapsulants with a low degree of cross-linking is 12 h compared to samples with high degree of cross-linking with 8 h, the GC values of badly cross-linked EPE encapsulants are expected to saturate at higher extraction times than shown here [13].

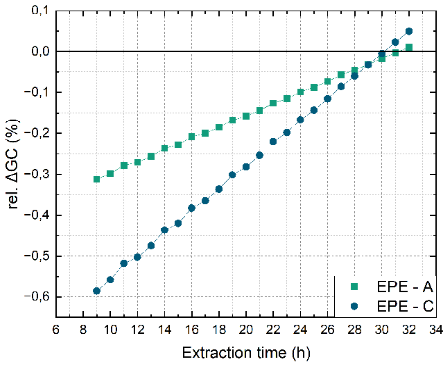

Figure 5: Calculated relative change in gel content of co-extruded EPE encapsulants as function of extraction time (3 samples per data point) using individually fitted curve data.

To further determine suitable process parameters for GC measurement of *POE-A* and *POE-B* using Soxhlet extraction the extraction time was varied similarly to both EPE encapsulants. The results of this extraction time sweep from 8-24 h can be seen in Figure 6 for both *POE-A* and *POE-B*. Both POE materials were laminated at 155°C for 20 min. These lamination parameters represent the longest recommended datasheet process.

POE-A exhibits high GC values starting at 87.35 % after 8 h extraction and decreasing to 82.48 % after 24 h with a total decrease of 4.87 % showing a similar behavior in GC changes compared to *EVA ref.* After 8 h extraction *POE-B* exhibits a measured GC value of 80.35 % decreasing to 73.16 % after 24 h with a total decrease in GC of 7.19 %. After 32 h extraction a change in GC of 0.1 % in case of *POE-A* and 0.02 % in case of *POE-B* can be measured in comparison to 24 h extraction. Since 8 h more extraction time leads to an insignificant change in GC for both POE encapsulants a complete extraction can be assured similar to the extraction time sweep for both EPE encapsulants.

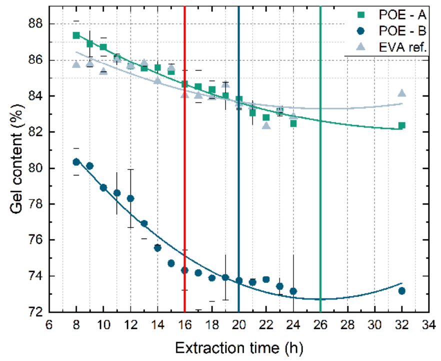

Figure 6: Measured gel content of POE encapsulants as function of extraction time (3 samples per data point) with fitted curves and calculated minimum extraction time (green & blue line) in comparison with recommended extraction time for POE [12] (red line).

The changes in GC values of both POE encapsulants are shown in Figure 7. Similar to the measured data of both EPE encapsulants in Figure 3 volatile jumps in GC values can be seen in case of both POE encapsulants. For graphical determination of the saturation point both POE encapsulants are individually fitted as shown in Figure 8 in case of *POE-B*.

The data of these individually fitted curves was then used to calculate the change in GC values of both POE encapsulants as seen in Figure 9. When analyzing for intersection with the 0 %-line *POE-A* exhibited saturation at 32 h of calculated extraction time with a GC value of 82.16±0.52 %, leading to a minimum extraction time of 26 h when considering standard deviation.

As seen in Figure 9 *POE-B* shows saturation at 26 h of calculated extraction time with a GC value of 73.17±0.52 % which corresponds to a minimum extraction time of 20 h. It should be noted that the influence of specific material on the minimum extraction time is more noticeable in case of POE with a difference of 6 h between *POE-A* and *POE-B* compared to a difference of 2 h with *EPE-A* and *EPE-C* as shown in Figure 2 and Figure 6 with vertical lines. This dependance on the specific POE encapsulation material was already mentioned by *Öz et al* [12].

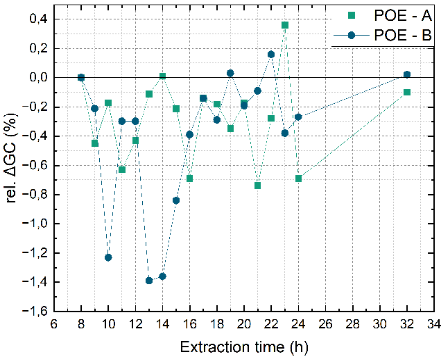

Figure 7: Relative change in measured gel content of POE encapsulants as function of extraction time (3 samples per data point).

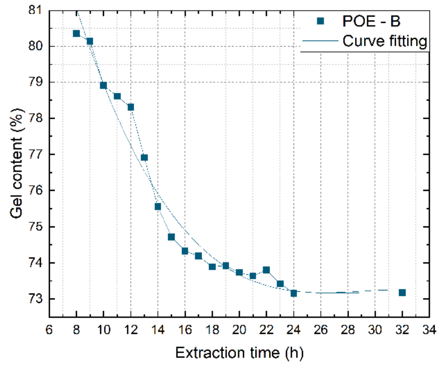

Figure 8: Measured gel content of *POE-B* as function of extraction time (3 samples per data point) with fitted curve.

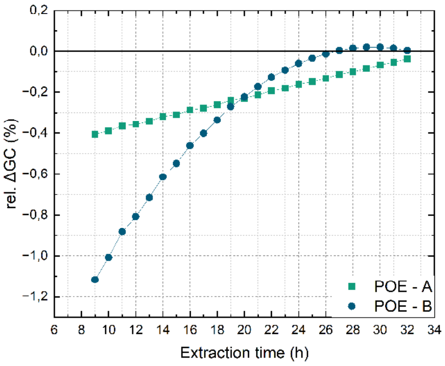

Figure 9: Calculated relative change in gel content of POE encapsulants as function of extraction time (3 samples per data point) using individually fitted curve data.

3.4 Mini-module reliability (*EPE-A* & *POE-A*)

In Figure 10 the power losses of mini-modules comprised of *EPE-A* and *POE-A* after different reliability tests are shown. DH1000 and TC200 tests resulted in average below -1.15 % power loss at MPP (*Maximum Power Point*) with the highest loss of -1.73 % in case of *DH1000_2*. After UV exposure the modules initially show power losses of up to -10 % in case of *UV60_1*. After stabilization (*stab_1*/stab_2, see Figure 10) by light soaking the modules show an average of -3 % in P_{mpp}. This so called *dark storage effect* is known for TOPCon modules and can be recovered by a light soaking process just prior to the power measurement as reported by *Gebhardt et al.* [14].

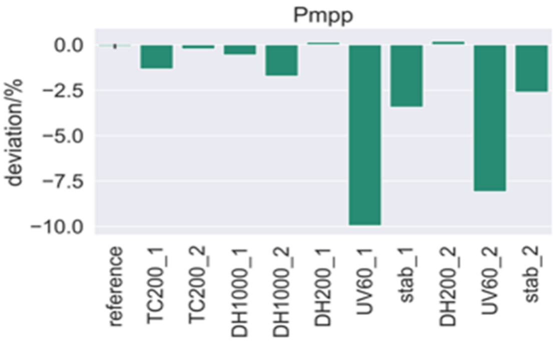

Figure 10: Power loss (ΔPmpp) of mini-modules (A-series encapsulants) after reliability tests.

In Figure 11 the captured EL images before and after DH1000 are shown. No significant visible damage can be observed which is consistent with the low power loss of -1.15 % in average.

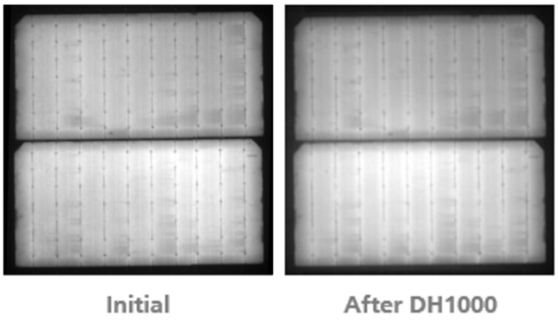

Figure 11: EL images of mini-module (A-series encapsulants) before and after DH1000.

4 CONCLUSION AND OUTLOOK

This study recommends suitable process parameters for Gel Content determination using Soxhlet extraction for co-extruded EPE. By systematically varying process parameters like extraction cycle time, number of extraction cycles and total extraction time a recommendation can be given for complete extraction of EPE and POE encapsulant films with a high degree of cross-linking. The point of complete extraction is determined as the extraction time at which the change in measured GC saturates. At this point an increase in extraction time will no longer have significant effects on the measured GC of the cross-linked encapsulant.

By variation of cycle time from 2-5 min in 1.5 min intervals the measured GC of *EPE-C* was analyzed regarding changes in GC values. Since decreasing the cycle time from 5 min to 2 min resulted in only - 0.43 % change in measured GC it can be concluded that the variation of cycle time in the tested range has no significant influence on the measured GC. Further testing showed that an increased number of extraction cycles and therefore a higher total amount of extraction time led to lower GC values. Due to this increase in extraction time more non-cross-linked compounds are being washed out resulting in a decrease in measured GC.

For testing the influence of resting time in the cooling solvent samples of *EPE-C* were taken out of the Soxhlet apparatus after different amounts of resting time after a finished extraction. Between the samples taken out after + 45 min and + 3 h of resting time a decrease of 1.39 % in GC was measured. Comparing the samples taken out after + 3 h and + 18 h of resting time no more significant changes in GC could be measured. This is explained by the remaining high temperature of the solvent and therefore higher diffusion coefficient allowing non-cross-linked compounds to diffuse out until the solvent cools down. After cooling down the diffusion process slows down heavily resulting in no more significant changes in measured GC. Due to this effect the resting time in the cooling solvent is highly important to accurately measure GC values. When being inconsistent with this parameter the results can become highly distorted and will no longer be reproducible.

To determine the minimum extraction time for EPE and POE with a high degree of cross-linking a sweep from 8-24 h extraction time was performed to analyze the change in GC for the point of saturation where a further increase in extraction time no longer significantly affects the measured GC. For *EPE-A* this point of saturation was found at 31 h (GC of 90.34±0.52 %) while *EPE-C* saturates at 30 h (GC of 85.70±0.52 %). Taking the standard deviation of 0.52 % into account yields a minimum extraction time of 22 h and 24 h for *EPE-A* and *EPE-C* respectively. The necessary extraction time needed for EPE samples with a low degree of cross-linking are expected to saturate at higher extraction times due to a higher percentage of non-cross-linked compounds needed to be washed out. Analyzing the POE samples, it was found that *POE-A* reaches saturation at 32 h (GC of 82.16±0.52 %) with a minimum extraction time of 26 h. *POE-B* exhibits saturation at 26 h (GC of 73.17±0.52 %) with a minimum extraction time of 20 h.

The tested mini-modules with *EPE-A* and *POE-A* encapsulants exhibited high stability with power losses of -1.15 % in average after DH1000 and TC200 tests and no visible damage in EL images. Initial UV-induced losses recovered after light soaking, stabilizing at around -3 % in average.

In further experiments the GC measurements for both POEs and EPEs conducted in this study will be analyzed in case of low degree of cross-linking. Since the recommended extraction time for EVA encapsulants differs from 8-12 h depending on the degree of cross-linking according to *IEC 62788-1-6* the GC values of EPE encapsulants with a low degree of cross-linking are expected to exhibit a similar behavior due to the multilayer EPE encapsulant being comprised partly of EVA encapsulant films [13]. With this a better understanding of the correlation between GC and necessary extraction time can be achieved.

5 ACKNOWLEDGEMENTS

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