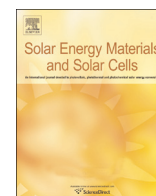




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# Determination of the degree of ethylene vinyl acetate crosslinking via Soxhlet extraction: Gold standard or pitfall?



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## ARTICLE INFO

## Article history:

Received 18 February 2015

Received in revised form

21 July 2015

Accepted 31 July 2015

## Keywords:

Photovoltaics

Ethylene vinyl acetate

Degree of crosslinking

Soxhlet extraction

Round-robin test

## ABSTRACT

Since the beginning of PV module production, Soxhlet extraction has been the standard method for the determination of the gel content and the resulting calculation of the degree of crosslinking of the most common PV encapsulant ethylene vinyl acetate (EVA). While the method is deemed well established and several pertinent ASTM and ISO standards exist, in practise the actual procedures used in – and trusted by – both industry and R&D institutions vary substantially. To evaluate the reliability of the methods and the comparability of the results, a round-robin test involving seven independent European laboratories – one industrial PV module manufacturer and six R&D facilities – was conducted. The measurements were performed using homogenous, anonymized sample sets, each comprising five differently crosslinked EVA foils. The analysis showed that results obtained for the same samples may deviate significantly, but also that very different analytical procedures can yield comparable values. In a systematic study, the impact of various key parameters of the analytical process (extraction time and solvent, drying conditions, sample size and weight etc.) was investigated. Based on these findings, deviations observed in the round-robin study could be linked to their origins and the main pitfalls were identified. In conclusion, a suggestion for an optimised standard procedure was derived to ensure comparable results at all laboratories.

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## 1. Introduction

Improving quality control in photovoltaics (PV) manufacturing became a major topic over the last years, especially since nowadays PV module manufacturers have to optimise system costs and warrant operational product lifetimes of 25+ years at the same time. The polymeric solar cell encapsulant is a key component in

this, since it decisively affects both the production effort and the operational lifetime of PV modules. PV encapsulants have to fulfil several fundamental functions over the entire operational lifetime of the module [1]: (i) mutual connection of the module components, (ii) structural support and mechanical protection of the solar cells to prevent over-stressing and cell cracking, (iii) prevention of the ingress of ambient media (humidity, oxygen etc.) and (iv) optical coupling of incident solar radiation with the solar cells. Selection and processing of the encapsulant hence vitally influences both, the long-term durability and the in-use performance of PV modules. Although various alternative materials have been introduced recently, at present crosslinked ethylene vinyl acetate (EVA) is still the by far dominating encapsulation material for PV modules.

PV-grade EVA is a random copolymer of ethylene and vinyl acetate, with a typical vinyl acetate percentage of 28–33% (w/w). During module lamination, the initially thermoplastic EVA is

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“cured” into a three-dimensionally crosslinked elastomer by a thermally induced radical crosslinking reaction [2]. The original mildly opaque, soft and easily plastically deformable EVA sheet is thus transformed into an elastomeric, robust and highly transparent encapsulation. Practical experience showed that a degree of crosslinking of  $\geq 70\%$  is required for PV applications to ensure high and constant product quality. To warrant the required quality and operational lifetime of PV modules, the degree of EVA crosslinking has to be routinely monitored in PV module production. Although some alternative analytical approaches for determining the degree of crosslinking of PV encapsulation have been demonstrated [3], including such that are rapid, non-destructive and potentially suitable for in-line implementation, based e.g. on mechanical nano-indentation [4] or Raman spectroscopy [5,6], the standard method is and remains the determination of the gel content by chemical solvent extraction at elevated temperatures, frequently referred to as “Soxhlet extraction”.

Since the beginning of industrial PV module production, this standard procedure is in use for determining the degree of crosslinking of EVA. It is based on the following steps: (i) measuring the weight of a specimen ( $M_1$ ), (ii) extracting the specimen with a suitable eluant to leach any extractable components, (iii) drying the residue to constant weight, (iv) determining the mass of the insoluble residue ( $M_2$ ) and (v) evaluating it against the initial specimen weight. The ratio of the mass of the extraction residue ( $M_2$ ) divided by the initial mass of the sample ( $M_1$ ) yields the “gel content” or Soxhlet degree of crosslinking

$$X \text{ (Soxhlet) [\%]} = \text{Gel content [\%]} = \left( \frac{M_2}{M_1} \right) \cdot 100, \quad M_2 \leq M_1. \quad (1)$$

Even though it is slow, involves hazardous chemicals and requires sampling, this absolute, comparatively simple and well-established extraction and gravimetric principle is the standard reference for all newly developed systems. A major practical problem, however, is that no dedicated, unambiguous international standard exists for the analysis of elastomeric EVA, PV-grade or otherwise. The closest applicable standards are ASTM D2765 [7] and EN ISO 10147 [8], both of which are designated to measuring the gel content of crosslinked ethylene plastics, typically tubes, fittings and electrical insulations, and the recently released EN ISO 6427 [9], which defines a general method for determining the amount of extractable components in plastics. Several of the parameters and parts of the sample pre-treatment procedures, however, described in these standards are not relevant or useful for soft EVA materials.

In the absence of a specific, obligatory standard, EVA analytics are routinely carried out by PV manufacturers and R&D laboratories is typically based on a (loose) derivative of one of these three standards. These standards allow a choice between two fundamentally different analytical procedures: EN ISO 10147 is based on immersion of the sample in a boiling solvent in a reflux arrangement,

ASTM D2765 also allows this analytical approach or the use of a Soxhlet extractor (stating in a comment that the Soxhlet approach is preferable in respect to both, accuracy and precision), and the most current standard EN ISO 6427 requires the use of a Soxhlet extractor or a comparable device, e.g. a Twisselmann extractor. Once having chosen the type of extractor, the standards also allow for setting a number of decisive experimental parameters according to preference and need, like e.g. the eluant and the extraction time. A comparison of the key analysis parameters according to the three standards is given in Table 1.

The inherent procedural variability of the standards causes a certain ambiguity, creating a known problem in particular when attempting to evaluate and benchmark data from different laboratories and manufacturers. To address this issue, a round robin-style inter-laboratory proficiency test for the determination of the degree of crosslinking of EVA derived from gel-content measurements by solvent extraction was initiated and conducted in a joint effort involving seven established European institutions that routinely analyse polymers and PV-grade EVA samples using established standard procedures.

According to the approved procedures of a method-open round-robin test, a set of identical test samples varying only in the state of curing was prepared by the study coordinator under controlled, standardized conditions. The samples were analysed for consistency and homogeneity using an independent spectroscopic method (Raman) and distributed to all partners in anonymised form. Each participating partner analysed the samples using its own established analytical procedures, and reported both, the results and the relevant procedural details back for further analysis. In addition, a systematic impact study regarding the key analytical parameters of a Soxhlet-type analysis was conducted to provide a sound basis for explaining possible deviations and eventually suggests a dedicated, reliable best practise procedure.

## 2. Materials and methods

Irrespective of the PV module manufacturer, the design of commercial PV modules is very similar: a solar glass front, two polymeric encapsulant layers embedding the solar cells and their electrical interconnects between them, and either a polymeric backsheet or another glass pane as back cover. These components are assembled, aligned and laminated using a combination of heat and pressure. The degree of crosslinking is controlled by the lamination parameters, primarily the lamination temperature and the crosslinking time, but it also depends on the composition of the EVA material, especially the type and initial concentration of the radical crosslinking agent. For the purposes of this study, only one type of EVA material was used. The experimental design for controlling the state of curing followed the industrial practise of

**Table 1**

Comparison of key analysis parameters for crosslinked EVA gel content determination, according to the different pertinent standards.

	ASTM D2765	EN ISO 10147	EN ISO 6427
Solvent	Decahydronaphthalene <i>or</i> xylene isomer mixture	Xylene isomer mixture	Xylene isomer mixture
Extraction procedure	Immersion extraction <i>or</i> Soxhlet extractor	Immersion extraction	Soxhlet extractor <i>or</i> similar
Test specimens	$0.300 \pm 0.015$ g; $\geq 2$ repeat determinations	$\geq 0.200$ g; $\geq 2$ repeat determinations	$\geq 1.000$ g; $\geq 2$ repeat determinations
Test specimen container	120-mesh (0.125 mm) stainless steel wire pouch	125-mesh (0.12 mm) stainless steel <i>or</i> aluminium wire pouch	Cellulose extraction thimble, stainless steel wire pouch, sintered glass crucible, 40–100 $\mu\text{m}$ pore size
Extraction time	6 h in decalin <i>or</i> 12 h in xylene	8 h $\pm$ 30 min	8 h at 15–25 cycles/h
Drying conditions	150 °C at $\leq 95$ kPa	90 °C at $\leq 85$ kPa <i>or</i> 140 °C at normal pressure	140 °C at normal pressure

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