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DETERMINATION OF EVA CROSS-LINKING DEGREE AFTER LAMINATION PROCESS BY EXTRACTION AND OPTICAL TRANSMISSION MEASURING

The ethylene vinyl acetate (EVA) is widely used for solar modules encapsulation. During lamination process EVA melts and chemical bonds between polymer chains are created. Its number is tightly related to cross-linking degree and it is consider as a major quality reference for module encapsulation. The lamination can be described as a process with two stages: melting and curing where the typical temperature for curing is in the range from 145 to 175°C. In the present study, for the first time, comparison of three commercial available EVA foils with low curing temperature EVA (EVA LOW). For this reason, the temperature of following lamination processes was set from a range from 115 to 175°C. The behavior of cured EVA films under investigation EVA was determined with two approaches: with extraction and with optical methods. The results indicate the applicability of these methods for the EVA cross-linking characterization. Finally, the extraordinary behavior of EVA LOW foil was noticed.

Keywords: Photovoltaics, silicon solar cells, lamination, gel content test, cross-linking degree

1. Introduction

To provide long lifetime of photovoltaic modules a proper encapsulation is mandatory. One of the most widespread method is lamination in a glass-glass system which highly protects solar cells from breakage or influence of atmosphere. Furthermore, it constitutes electrical isolation for a solar system [1]. The factor which joins whole set together and enables a proper stabilization of the solar cells is ethylene-vinyl acetate (EVA). EVA is multitasking, copolymer sheet, which becomes transparent during lamination process. To provide high adhesion to the glass, EVA is modified by adding adhesion promoter, which enables bonding between the glass to back sheet and curing agent [2-4]. The cross-linking of polymer is a process where spatial (rubber) structure is created due to the bridges formation in the form of covalent or ionic bonds among polymer chains. Polymer reacts with the curing agent and the reaction is initiates by temperature elevation, pH, pressure or irradiation. After this process, polymer starts to be insoluble and only swells due to solvent sorption. Moreover, its flexibility decreases while hardness and melting temperature increases. To eliminate water and air from the module, as the potential reason of further delamination whole process is performed in vacuum [5]. The cross-linking of EVA is significant and it has a major impact on set stability. Therefore, curing degree is a valid parameter and it carries information about the quality of laminated system. To determine the curing degree, different methods like extraction [3,6,10], optical measurement [3,7], Raman Spectroscopy [8], FTIR [5], Differential Scanning Calorimetry (DSC) [3,5,7] or Electron Beam Irradiation [9] can be applied. The gel content above 80% determines right mechanical and UV protection [3].

In spite of the fact, that encapsulation process contributes in significant way to the proper stability of the solar modules, only a little attention was paid to proper EVA selection and its characterization. To fill this gap a systematic investigation focused on comparison of the different EVA foil were undertaken and the results were presented in this paper. Therefore, the aim of this investigation was deep characterization of EVA foil behavior and comparison of cross-linking degree obtained by using different evaluation methods for commercially available EVA foils.

2. Experimental

2.1. Lamination process

The lamination process of glass-EVA-glass systems was performed in a flat-bed laminator P-Energy L200A. The peak temperature of lamination was located between 115 and 175°C. Time and pressure during process were the same for all the EVA samples. The process is typically performed in two steps – melting and curing phases. Three commercially available EVA foils,

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denoted as: EVA 1, EVA 2, EVA 3 and one low temperature EVA, denoted as EVA 4 (LOW) were tested for the lamination. Peak temperatures of curing phase were set to 115, 125, 135, 145, 155 and 175°C and this temperature was confirmed by a thermocouple and compared to the results from thermographic camera. The temperature distribution on laminator table is showning in Fig. 1. The maximum deviation was about 4°C, but the samples were placed on the surface of the same temperature and maximum deviation during lamination process is on a level of 1°C. The specimen between glasses after lamination was approximately 0.5 mm.

2.2. Extraction

Extraction is a simple separation method where pieces of laminated EVA foil are subjected to dissolution performed in Soxhlet set or in the bottle with heating for at least 24 hours. Over this time, non-cured, polymer residuals are spread out while cross-linked part remains undissolved.

A gel contents from different lamination conditions and various EVA foils were designated by using toluene solution. 1g of laminated EVA sheet was collected and dissolved in 100 ml of toluene (CHEMPUR, purity 99.7%) with addition of 0.0865g BHT (2,6-di-tert-butyl-4-methylphenol) (ACROS, purity 99.8%) at 60°C for at least 24 hours. Next, EVA gel was filtered and dried again at 105°C for more than 4 hours. Subsequently, the samples were weighted and insoluble weight fractions were calculated according formula:

$$\frac{m_3 - m_2}{m_1} * 100\% = \%Crosslinked\ material$$

where:

- m_1 – weight of the original specimen,
- m_2 – weight of the weighting bottle, cover and dried filter paper,
- m_3 – weight of the weighting bottle, cover, dried filter paper and dried residue.

2.3. Optical measurement

According to the literature, the optical measurement can be used as a nondestructive EVA cross linking estimation [3]. Therefore, transmission (T) and diffuse transmission (Td) throughout the glass-EVA-glass system were measured. Lamination set, consisted of two pieces of glass size 5 cm × 5 cm and EVA foil, constituted a sample to study. The lamination (curing phase) time was 460 seconds and applied temperature was within the range of 115°C-175°C. The optical measurements were conducted using UV-VIS-NIR Perkin Elmer Spectrophotometer Lambda 950 S in the range of 250-1000 nm for T and R and 300-650 nm for Td. The low value of the end of the range (1000 nm) was set because the external quantum efficiency of silicon solar cells above 1100 nm is below 30% [11]. To determine correlation between gel content test results and transmission of glass-EVA-glass system the haze factor (H) was calculated. The coefficient is a ratio of diffuse transmission and total transmission.

3. Results and discussion

3.1. Extraction

It was highlighted above, that the proper solar module encapsulation requires at last 80% of cross-linking of EVA foil. As presented in Fig. 2 more than 80% of EVA was cured at 135°C for EVA 4 LOW, both EVA 1 and EVA 2 achieved such a level when cured at 145°C and EVA 3 needed 155°C. The highest value of gel content for EVA 4 LOW was 89%, 90% for EVA 1, 95% for EVA 2, and 93% for EVA 4. It is worth to notice that for EVA 4 LOW cross-linking degree exceed 70% for such a low temperature as 125°C. When filtering of toluene – EVA 4 LOW mixture heated to 115°C, ductile gel was noticed, what has not been observed for other EVA samples. Extraordinary behavior of EVA 4 LOW comes from its modification by additional curing agent.

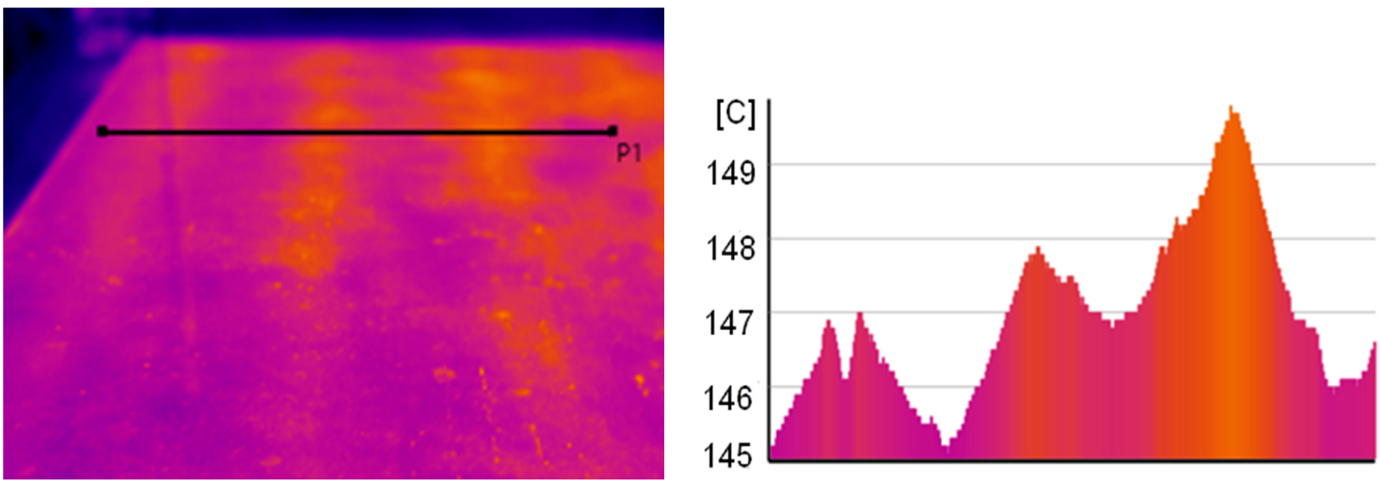

Fig. 1. Temperature deviation on the laminator table

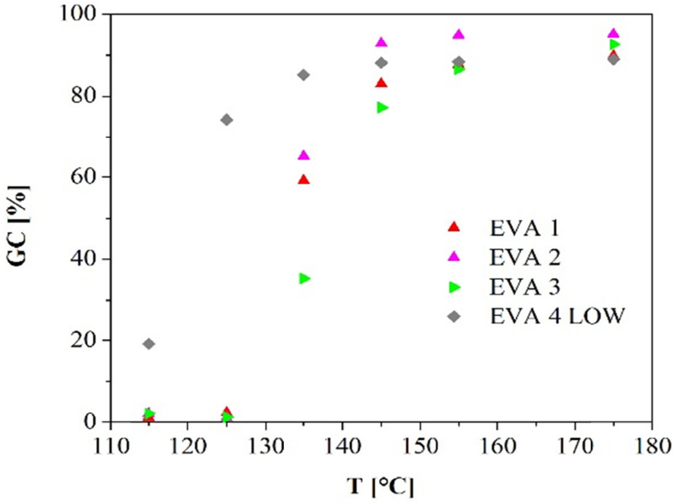

Fig. 2. Gel content test results from EVA foils after lamination process carrying at various temperatures

3.2. Optical method

The optical transmission was performed for glass/EVA/glass layouts where 115°C, 125°C, 135°C, 145°C, 155°C and 175°C lamination temperatures were applied. Figure 3 shows transmission plots for the EVA 1, EVA 2, EVA 3 and EVA 4 LOW, while Fig. 4 presents diffuse transmission plots for these samples.

The highest value of transmission was noticed for glass/EVA 2/glass system laminated at 155°C, which corresponds to 95% of gel content indicated by the extraction method. It should be pointed out, that this result was a maximum amount of gel content for the whole investigated EVA foils. In the wavelength range from 400 nm to 750 nm, results show that system transmission increases when non-cured residuals in EVA vanishes. Moreover, set with EVA 4 LOW exhibits transmission shift as a result of the higher absorption in the

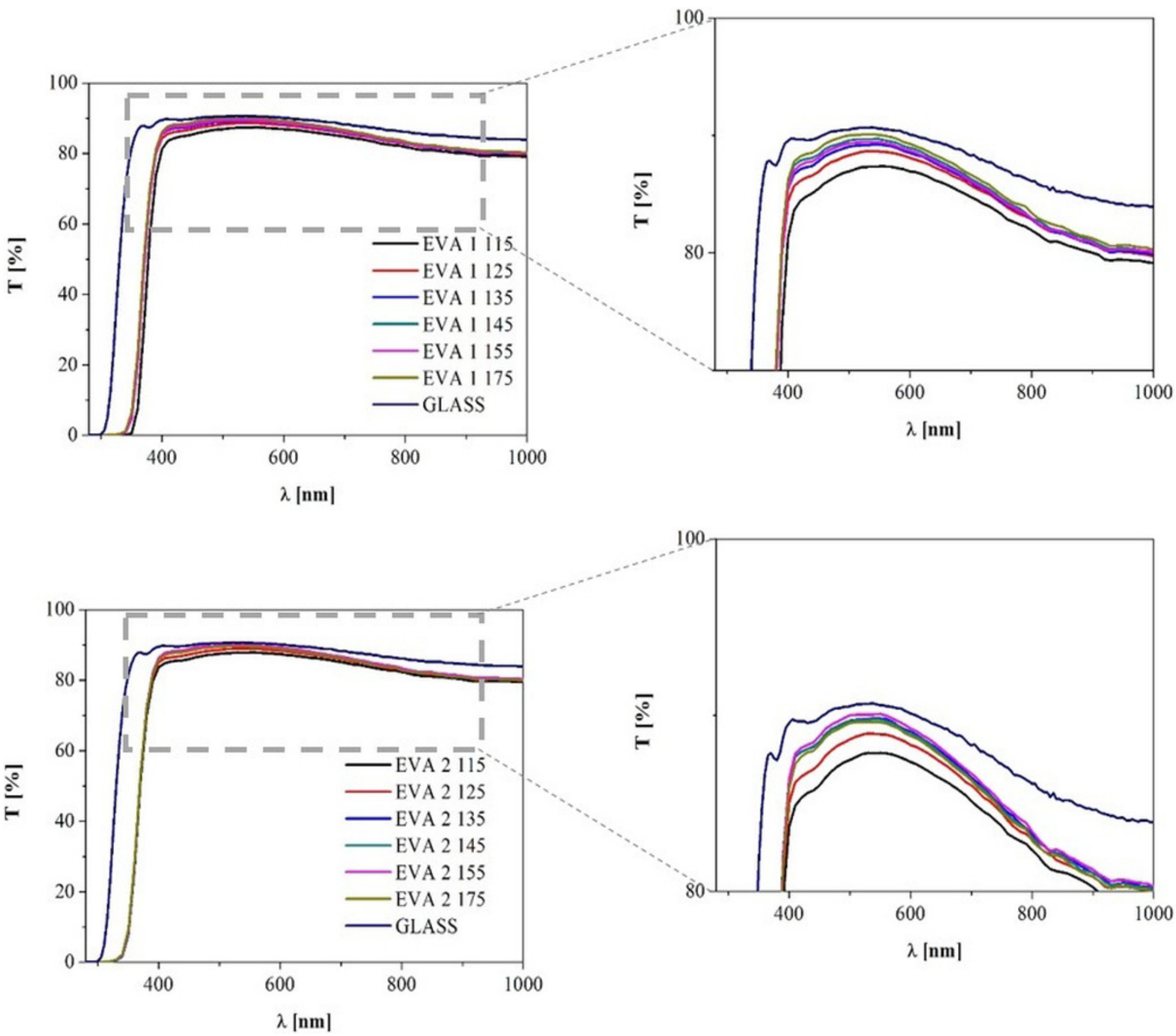

Fig. 3. The transmission plots for glass/EVA/glass systems with different EVA foil laminated in various temperature

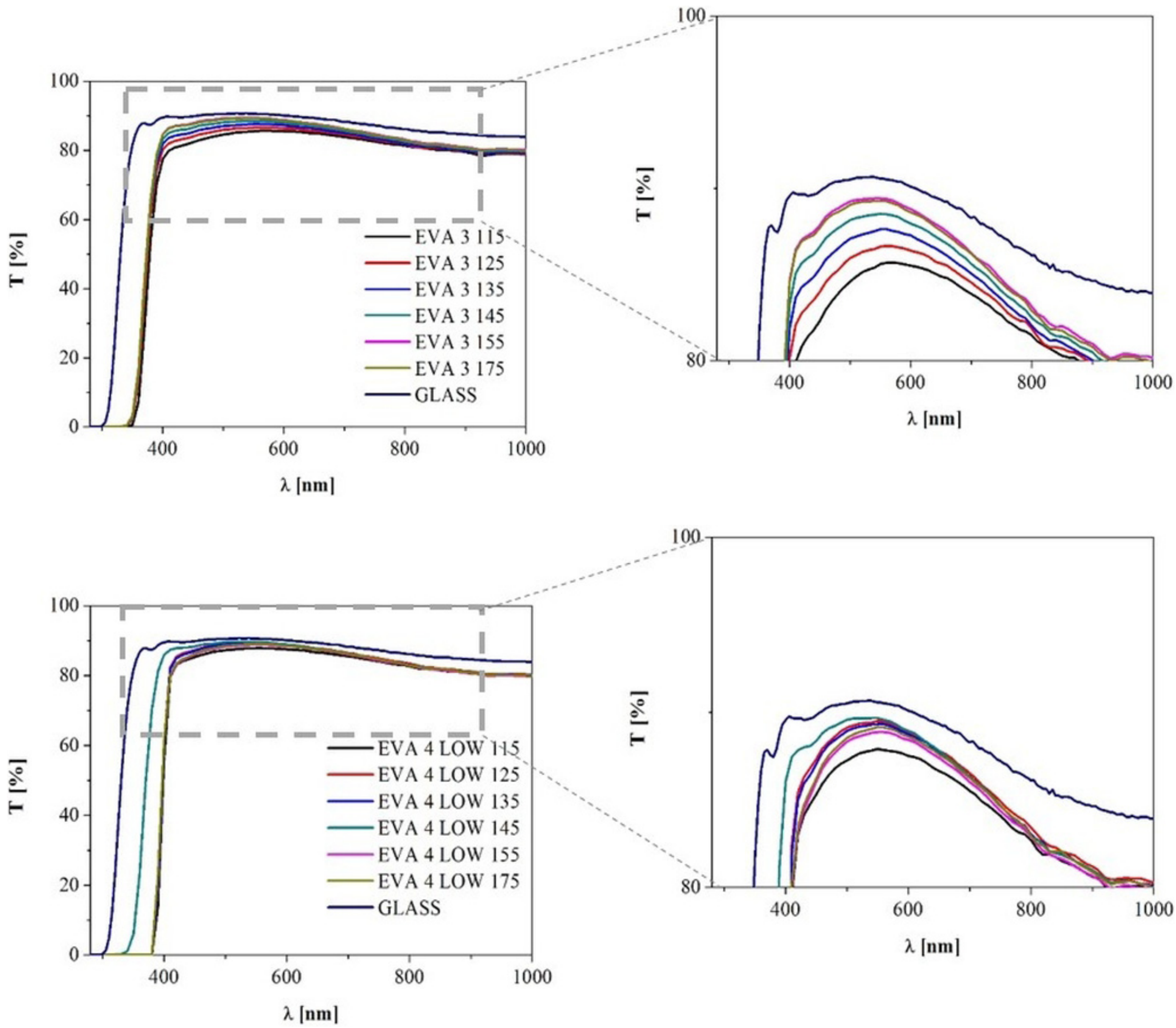

Fig. 3 Continued. The transmission plots for glass/EVA/glass systems with different EVA foil laminated in various temperature

short wavelength region. For EVA 4 LOW 145 the radiation cut-off region was located in the wavelengths shorter than 330 nm while for another EVA 4 LOW it was 380 nm. No such discrepancies were observed for another EVA systems where absorption edge was found at 340 nm for EVA 3 and 345 nm for EVA 1 and EVA 2. It can be expected that additives, which make EVA 4 LOW appropriate for the low temperature lamination, exhibits high absorption in this region and they are a cause of plot shift.

For all the samples, diffuse transmission was at the highest level for foils laminated at 115°C. The maximum T_d was noticed for EVA 1 (13.9%) and it was observed at 400 nm. Also for EVA 3 the maximum value of T_d was located at 400 nm (12.6%). While, for EVA 2 and EVA 4 LOW the greatest diffuse transmission was observed respectively at 390 nm (11%) and 410 nm (6%). It should be highlighted that the maximum of T_d for higher temperatures was not shifted for EVA 1 and EVA 3. Whereas, for EVA 2 and EVA 4 LOW it was moved towards longer wavelengths, respectively to 400 nm for EVA 2 and 420 nm for EVA 4 LOW. The shift of T_d plots for EVA 4 LOW is tightly related with transmission displacement and has the same ground. For EVA 2 it can be anticipated that this foil contains different curing agent than EVA 1 and EVA 3, therefore, its cross-linking was more effective with no transmission losses for short wavelength contrary to observed for EVA 4 LOW.

The haze factor (H) was determined as a ratio of T_d and T. H decreases when temperature rises and related gel content increases as well as presented in Fig. 5.

TABLE 1 shows gel content extraction results with corresponding haze factor. For EVA 2 and EVA 3 laminated at 115°C gel content test has shown higher value of cross-linking degree than for the same foils laminated at 125°C. It should be noticed that transmission for both sets laminated at 115°C was lower than for systems laminated at 125°C, moreover, T_d took

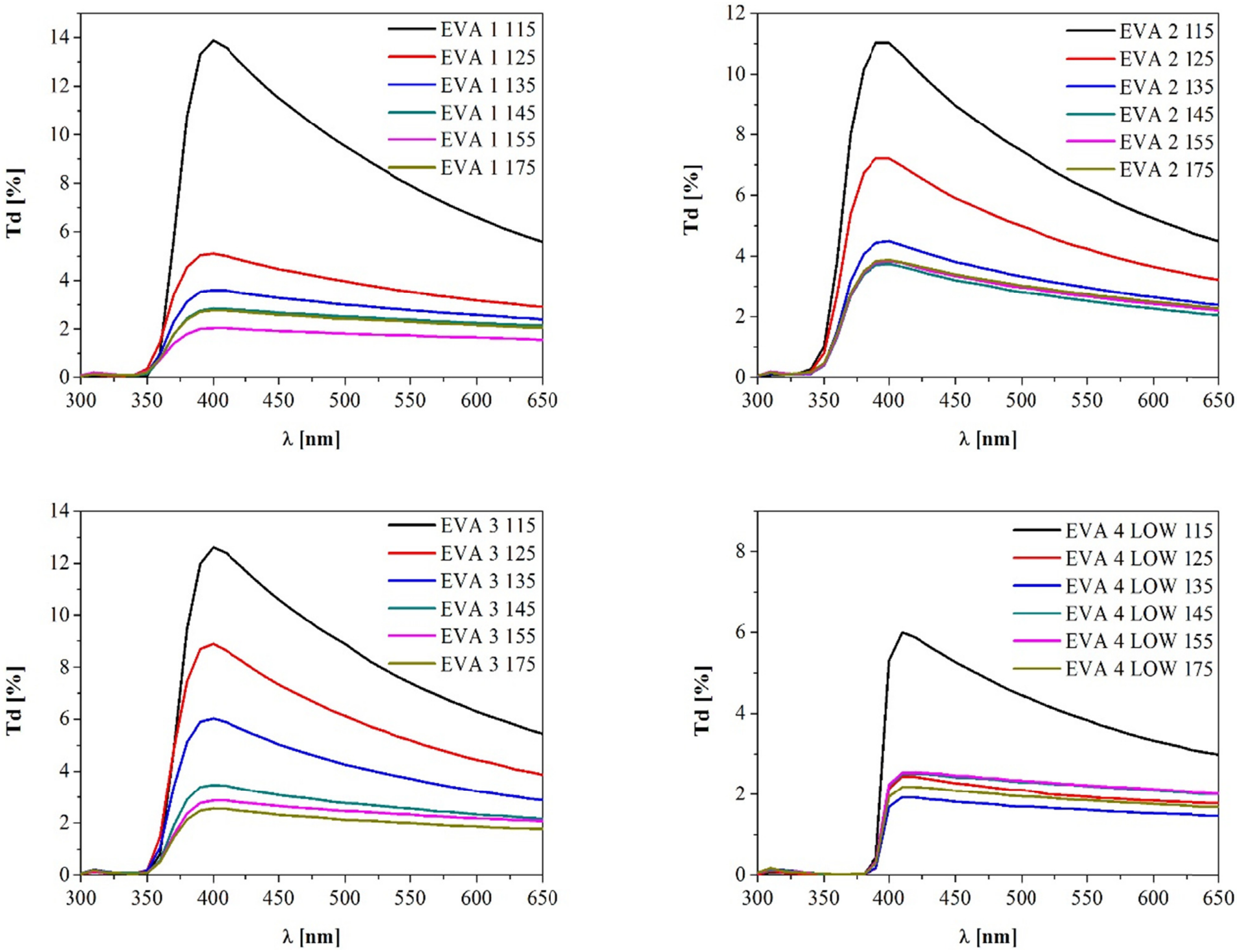

Fig. 4. The diffuse transmission plots for glass/EVA/glass layout

TABLE 1

Comparison of haze factor and Gel content test results

Lamination temperature [°C]	EVA							
	EVA 1		EVA 2		EVA 3		EVA 4 LOW	
	H [%]	GC [%]	H [%]	GC [%]	H [%]	GC [%]	H [%]	GC [%]
115	17.1	1.17	13.9	2.04	16.3	2.12	7.5	19.21
125	6.1	2.33	8.9	1.17	11.1	1.24	3.0	74.28
135	4.2	59.16	5.2	65.11	7.4	35.31	2.4	85.16
145	3.3	83.02	4.3	92.81	4.2	77.25	3.0	88.23
155	2.4	87.62	4.4	94.85	3.4	86.56	3.1	88.35
175	3.2	89.81	4.5	95.06	3.0	92.66	2.6	88.96

higher values for EVA 115°C. Due to the fact that the amount of residual gel was very low in both cases, any mistake made at any stage of the analysis could affect the final value. Consequently, discrepancy was considered as negligible.

The general correlation between gel content test results and haze factor is noticeable. The haze factor decreases when curing state goes up. Nevertheless, it is not possible to unambiguously determine how H is changing with cross-linking degree.

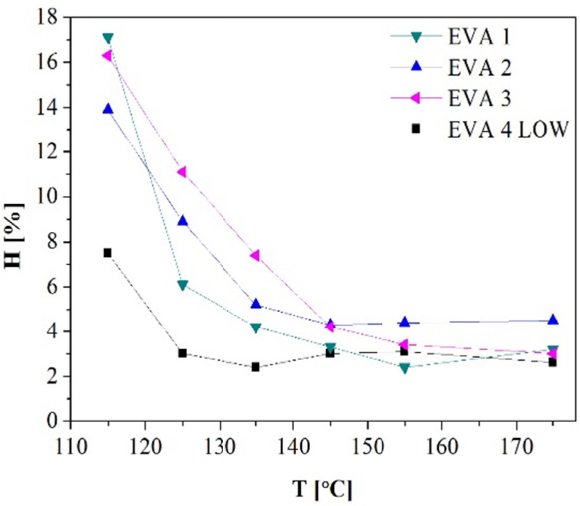

Fig. 5. Correlation between lamination temperature and haze factor of the light

4. Conclusion

Optical method, to cross-linking degree determination, shows good agreement with the result of extraction method. Therefore, optical measurement can be fast and nondestructive alternative for conventional gel content test. Moreover, it does not require to use hazardous toluene. However, the haze factor is individual parameter for each EVA foil and non-universal haze factor scale can be established.

The EVA 4 LOW achieved more than 70% of cross-linking degree at only 125°C which is unattainable for other EVA foils. Therefore, using low-temperature EVA 4 LOW in the solar cell lamination process can be beneficial from economical point of view. The EVA 2 reached the highest value of gel content (95%) without simultaneous transmission decreasing for short wavelength. Obtained results confirmed that EVA foils present different behavior and because of that lamination process has to be adjusted to selected foil.

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