SELECTION OF LED OR CONVENTIONAL UV LAMP BASED ON THE COMPOSITE THICKNESS

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Abstract

Composites matrices, in combination with a proper photoinitiator system, can be cured in much less time compared to thermal cured resins under the ultraviolet (UV) exposure, without losing or even improving the resulting mechanical properties. As the photopolymerization of thick composites is relatively difficult due to issues associated with the light absorbance through the material, a proper pairing of the emitting UV source and the photoinitiator is needed. This paper presents the comparison of the interlaminar mechanical properties of a glass-fibre/polyester composite cured with UV LED and conventional flood UV lamp light sources based on composite thickness. The interlaminar shear strength was higher for the UV LED light cured composites, being the highest differences as the specimen's depth increases, up to 10%. Likewise, the curing time is lower when the UV LED source is used.

1. Introduction

The high operational costs involved in combination with the intricacy of the manufacturing techniques currently employed have restricted wider industrial use of composites. For these reasons, considerable effort has been made to find and develop alternative cost-effective routes for manufacturing composite materials [1]. Some of such alternative routes are microwave curing [2], electron beam curing [3] and ultraviolet (UV) curing [4-7].

The UV curing industry, using the energy of UV light in the formation of polymeric materials, has approached the last years, highest degree of maturity. The development of monomers, oligomers, and photoinitiators during this time has allowed the technology to advance into very efficient formulations for a wide variety of applications [8]. Resins such as vinylester [5], epoxy [6] and polyester [7], when formulated with a proper photoinitiator, can be cured in minutes under exposure to UV light. The increased speed of cure possible through UV curing and the fact that the first part to cure is the component surface can also reduce the emission of volatile organic compounds (VOCs) from the resins [5]. Furthermore, as they have an indefinite pot life, the characteristics of the resin system allow complete impregnation of the fibre without premature gelation. This low-energy, cold cure, environmentally friendly technology has broken into conventional solvent-based resin technology and is now used in coatings, varnishes, graphic arts, high-speed printing, metal decorating, adhesives, etc. [8].

Although nowadays UV curing is more used in the covering industry, this curing method is getting more common in composite manufacturing. An example of the contribution of UV curing to the composite manufacturing industry is that the temporal control offered by this technology should alleviate many of the problems associated with Resin Transfer Moulding (RTM): first, allowing the mould to be completely filled with a low-viscosity resin before the start of the reticulation; and second, initiating the fast photopolymerization after the mould is completely filled. Other processes such as pultrusion and filament winding would be good applicants to combine with UV technology [9].

As UV energy can only penetrate optically transparent materials, and due to the absorption of radiation passing through matter [10], the thickness of laminates for efficient applications of UV curing is limited to a maximum depth of between 8 mm and 13 mm in one shot [4]. Therefore, new curing conditions, parameters and strategies would be taken into account since the curing of thin systems is completely different to the curing of a composite material. Photopolymerization of thick systems (of the order of 1 cm) is relatively difficult due to issues associated with the light absorbance through the material. However, some researchers have reported that efficient photopolymerization of thick polymers is possible if the emitting spectrum and photoinitiator systems absorbing spectrum are properly chosen [11]. Thus, proper pairing of the emitting UV source and the photoinitiator is considerably more complex in thick systems than in thin systems in which it is only necessary to ensure that the initiator absorbs at the most significant emission wavelengths.

UV bulb lamps are widely used in the industry. The emitting spectrum of those lamps is very wide, with lots of different intensity peaks and high presence of infrared radiation (IR), as the operating temperature of the bulb is between 600-800 ºC [8]. Nevertheless, UV LED sources present longer life time, less energy consumption, low heat generation, higher intensities and tighter emitting spectrum, with only one intensity peak, located at 365 nm or 395 nm depending on the UV LED source [11], allowing an efficient pairing of the UV source and the photoinitiator as the emitting spectrum is very narrow. Thus, as it is shown in the study carried out by Kenning [12] *et al.*, UV LED light sources may lead to more effective photopolymerization of thick polymer systems in addition to the advantages above commented.

This paper presents a comparison of the interlaminar properties of a glass-fibre/polyester composite cured with UV LED and conventional flood UV lamp light sources. Using each light source two laminates types were manufactured varying the sample depth: 3 and 6 mm. The same polyester resin was used with two different photoinitiator systems, each one specific for each light source.

2. Experimental

2.1 Materials

The material used in this study is a UV cured glass/polyester composite. The reinforcement consists of 300 g/m^2 quasi unidirectional E-glass ribbon. The reinforcement is described as quasi unidirectional because of the small proportion of fibres of 90º which maintain the cohesion of the unidirectional fibres. The resin was UV curable unsaturated polyester supplied by Irurena S.A. The lamps used are the followings: a 400 W flood type lamp provided by Dymax with an intensity of 75 mW/cm^2 in the UV range; and a UV LED source provided by Phoseon Technology with a maximum intensity of 8 W/cm^2 and the main emitting spectrum

peak is located at 395 nm. The emitting spectrums of the UV sources are presented in the Figure 1.

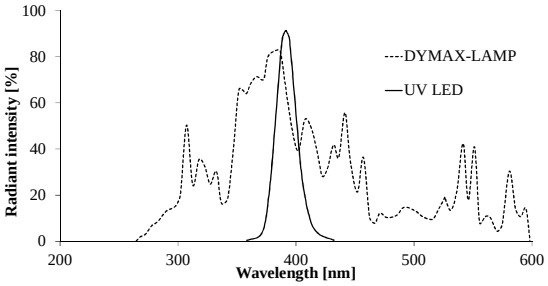
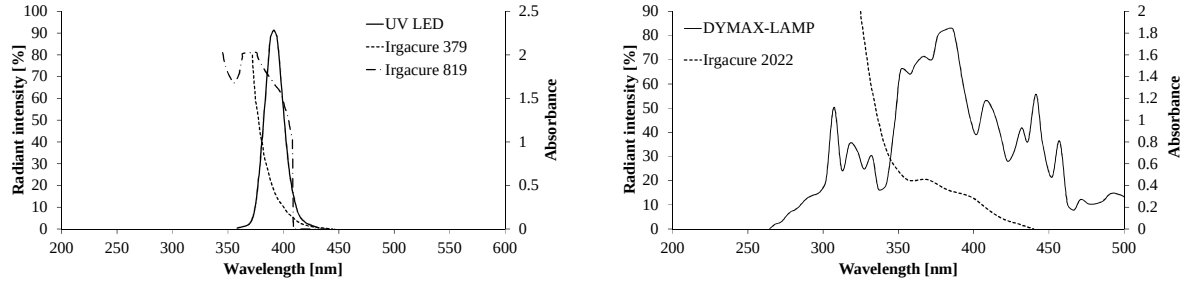


Figure 1. Emitting spectrums of the UV sources.

As the emitting spectrum of each lamp is notably different, two different photoinitiator systems were used. On the one hand, a system formulated with Irgacure 379 and Irgacure 819, which is designed for an efficient UV curing with UV LED; and in the other hand, a system formulated with Irgacure 2022, which it has been used in other studies carried out by the group with Dymax bulb lamp [12, 13]. The absorption spectrum of those photoinitiators is presented in Figure 2. Although the UV LED can provide higher values of exposure intensity, it has been limited to 1 W/cm^2 in order to maintain the same curing conditions for both sources. As the Dymax bulb lamp has an emitting area of 400 cm^2 and the power of the bulb is 400 W, the total intensity dosage for both sources is 1 W/cm^2. The main difference between both UV sources apart from the different emitting spectrums is that the UV LED's light is fully ultraviolet.



(a) (b)

Figure 2. (a) Photoinitiators for UV LED source. (b) Photoinitiators for UV bulb lamp.

Composite with two different thicknesses have been manufactured. The thinner one is a 12 layer laminate, whereas the thicker has 24 layers. All the specimens were manufactured by hand layup process. In order to maintain the repeatability of the thickness in all the specimens, a thickness plate has been used during the curing process as it is shown in the Figure 4. As it is said before, two different UV sources have been used in the fabrication of those specimens: UV LED and typical UV bulb lamp. Thus, 4 different types of specimen were manufactured (Table 1): two of them with 12 plies, one cured with the UV LED and the other one cured using the bulb lamp; and another two with 24 plies, cured in the same two ways. Two laminates of each composite type were manufactured.

Specimen	Number of layers	Thickness	UV source
3-LED	12	3.00 ± 0.03	LED
6-LED	24	5.94 ± 0.01	LED
3-BULB	12	3.04 ± 0.02	BULB
6-BULB	24	5.92 ± 0.02	BULB

Table 1. Manufactured specimens.

2.2 Test geometry and procedures

As the fibre-dominated tensile properties would present similar properties for each pair of specimen type, in order to determine the effect of the UV source in the curing process the interlaminar shear strength (ILSS) was selected, since the failure mode is matrix dependent. So as to determine the ILLS properties, the standard test method for short-beam strength has been used [14]. According to this method, the following specimen geometries were chosen (Figure 3b): on the one hand, the specimen length, *l*, should be six times the thickness, *e*; on the other hand, the specimen width, *b* should be two times *e*. Finally, the span length should be four times *e*.

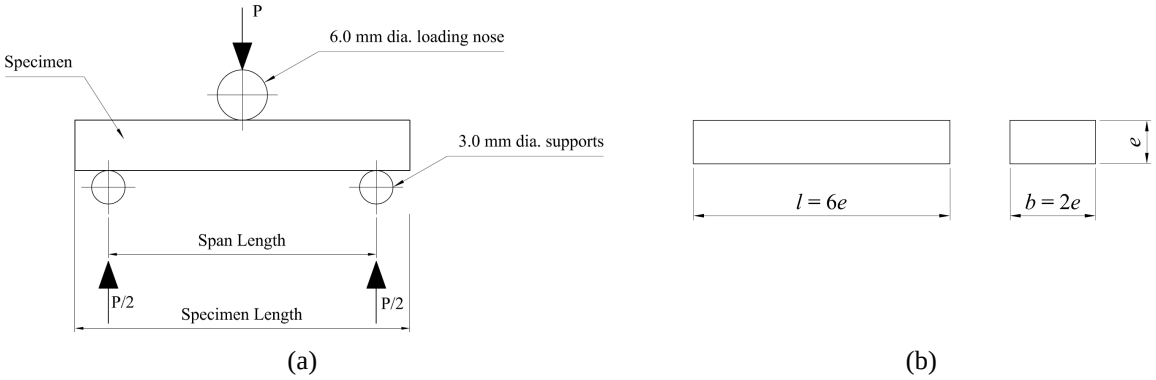


Figure 3. (a) Horizontal shear load diagram. (b) ILSS specimen configuration.

All tests were performed at displacement rate of 1 mm/min and using a 5 kN load cell. Five specimens of each type were tested. The short-beam strength is calculated following the next equation:

$$F^{sbs}=0.75\frac{P_m}{b\cdot e} \tag{1}$$

where, F^{sbs} is the short-beam strength (MPa), P_m is the maximum load observed during the test (N), *b* is the specimen width (mm) and *e* is the specimen thickness (mm).

Related to the curing analysis, a specific tool has been developed to control the curing process. The specimens where manufactured by hand layup process. Thus, after the impregnation of the fibres, the uncured specimens where placed into the curing tool. Figure 4 shows a description of the curing analysis tool, which permits to change the next main curing parameters: the UV source, the distance between the source and the specimen, the thickness of the specimen. The curing analysis is made by the control of the electric resistivity of the material in the non-exposed surface, which is the last area to cure.

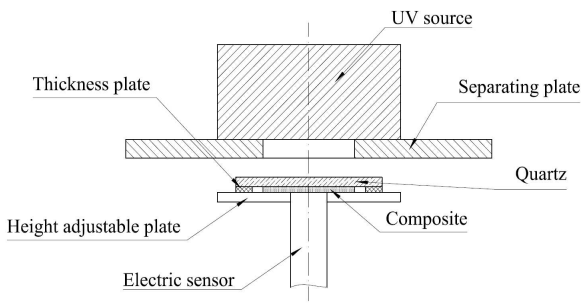
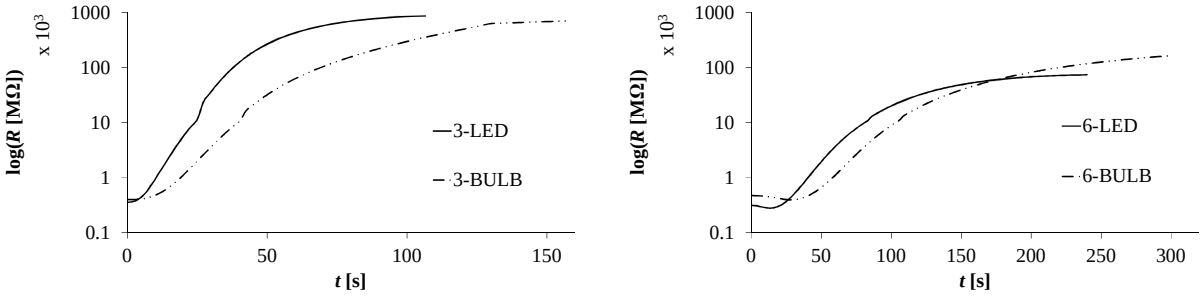


Figure 4. Description of the curing analysis system.

As it is shown in the figure above, the electric sensor is located in contact with the non-exposed surface, measuring the variation of the electric resistance of that area of the composite during all the curing process. So as to obtain a repeatable thickness for different specimens, some plates (thickness plates) were used between the quartz plate and height adjustable plate. The quartz (UV transmittance of 92 %) has been employed to compress the composite up to the thickness plate. The height adjustable plate permits different curing conditions by changing the distance between the UV source and the composite. In order to ensure that all the specimens are fully cured, the next statement should be satisfied [12]: the composite will be fully cured when there are not significant changes in the electric resistance and the hardness of the exposed and non-exposed surfaces are equal. The surface hardness was measured using a Barcol durometer, which is recommended for the use with composites. Additionally, the temperature of the composite will be measured at the non-exposed surface using the temperature sensor that is incorporated in the electric sensor.

3. Results and discussion

As it has been described previously, the electric resistance of the material was monitored during the UV curing process of all the specimens. Figure 5 shows the electric resistance measurements of the most representative manufactured laminates. Anyway, during the monitoring is has been noticed that the behaviour was comparable to the other laminate manufactured in the same conditions. Analysing the following resistance values, it can be noticed that all the specimens follow the same resistance curve pattern during the curing, and at the final state they reach a plateau which is related to the total curing of the specimens. Moreover, no significant differences of hardness were found between the exposed and non-exposed surfaces of all the specimens. Thus, this fact confirms that all the specimens where fully cured as it was observed in the electric monitoring.



(a) (b)

Figure 5. (a) Specimens of 3 mm thickness. (b) Specimens of 6 mm thickness.

The experimental curves for the conversion as a function of time can be obtained from the electric resistance curves previously presented. Moreover, deriving those curves, the experimental curves for the conversion rate as function of time can be obtained, as it reported in the Figure 6. First of all, analysing the conversion as function of time curves, it can be concluded that specimens cured by irradiation of the UV LED source reach the complete curing faster than the specimens cured with UV bulb lamp. The time difference in the case of the specimens with 3 mm thickness is about 50% more in bulb cured specimens, while curing time for composites with 6 mm thickness is 20% higher for the bulb lamps. This means that the UV LED source permits a faster curing process than the UV bulb lamp in the same curing conditions. This statement is confirmed if the conversion rate as function of conversion curves are analysed. In those curves it can be seen that the conversion rate for the same conversion is

always higher for the UV LED cured composites, which means that UV LED source allows a faster photopolymerization.

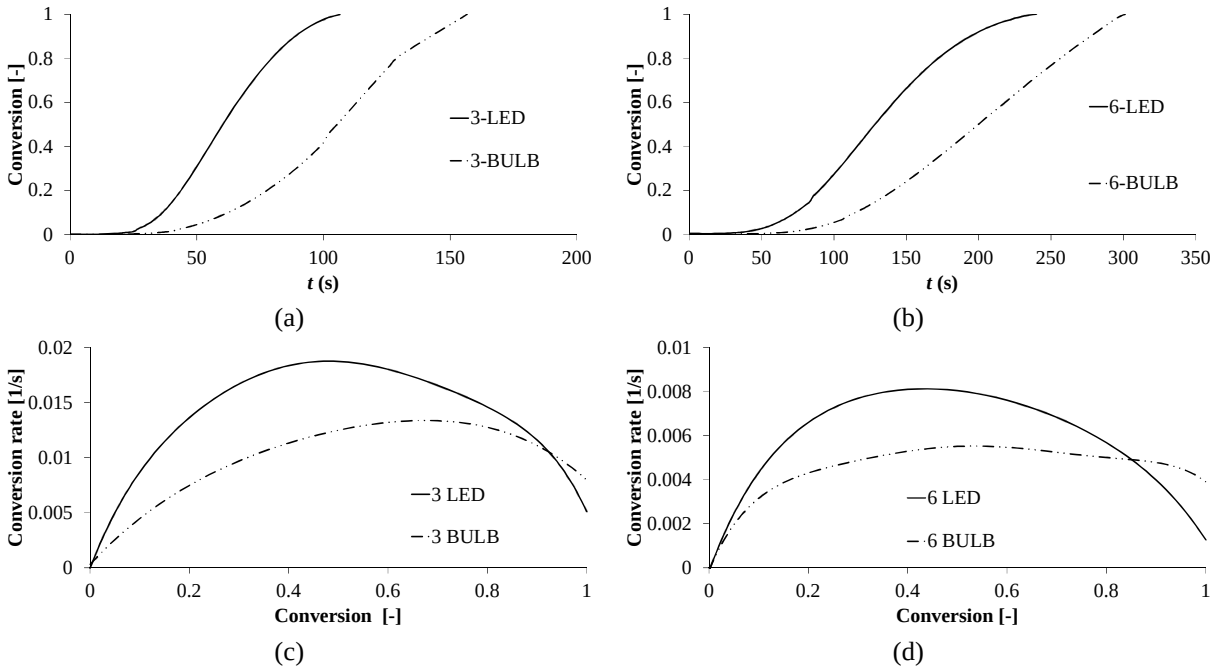


Figure 6. (a) Conversion vs. time – 3 mm thickness. (b) Conversion vs. time – 6 mm thickness. (c) Conversion rate vs. conversion – 3 mm thickness. (d) Conversion rate vs. conversion – 6 mm thickness.

Continuing with the analysis of the curing, the comparison of the curing time is needed in order to compare the curing behaviour for different thicknesses. The Figure 7 shows an analysis of the curing times in the previously presented conversion as function of time curves. On the one hand, it can be seen that the time needed by the photopolymerization front to achieve the non-exposed surface (t_{init}) is lower for UV LED cured specimens. Moreover, it is noticed that the t_{init} for the 6-LED and 3-BULB is similar. This fact is due to the high penetration capacity of the UV LED source, which can penetrate 6 mm of composite in a similar time than the bulb lamp in the case of 3 mm. On the other hand, the slope of the conversion (conversion rate) of the bulb cured specimens is lower than the LED cured specimens for both thicknesses. Those factors have direct influence in the resulting total curing time.

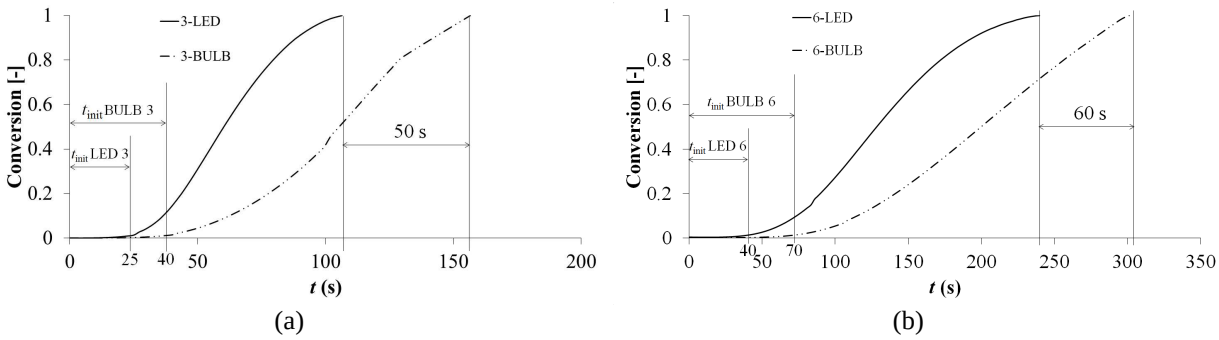
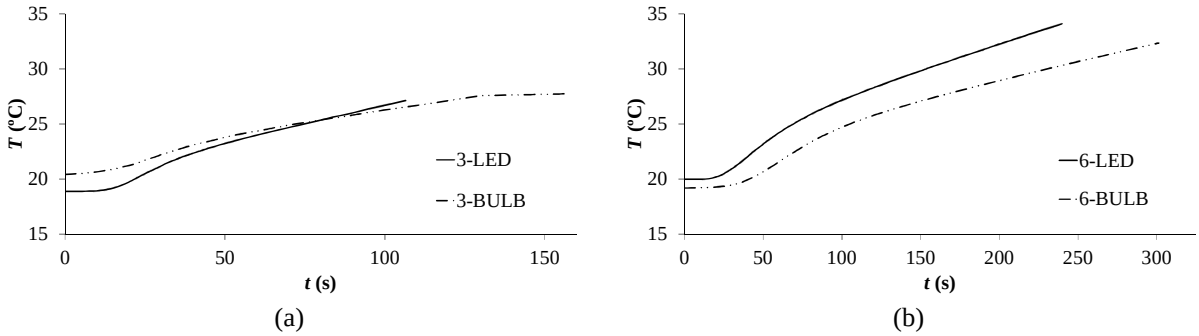


Figure 7. Curing time comparison.

The comparison of the measured temperature at non-exposed surface is reported in Figure 8. No significant differences have been noticed during the curing process.



(a) (b)

Figure 8. Comparison of the temperature at non-exposed surfaces.

In order to determine if the failure of the tested specimens was interlaminar failure, all the specimens were analysed in a macroscope after ILSS tests. All the specimens present the same interlaminar failure mode after the short-beam test. After that it can be concluded that the values obtained for those specimens can be compared as the same failure pattern is found in all the specimens. Short-beam strength, F^{sbs}, determined from short-beam test for 4 type of specimen manufactured is shown in Figure 9. Interlaminar shear strength was higher for the UV LED light cured composites, being highest differences as the specimen's depth increases. A highest deviation is noticed in composites with 6 mm thickness. Anyway, the same deviation was found in both types of specimens. It can be concluded that using UV LED sources maintain the interlaminar properties of the composites comparing with UV bulb lamp cured composites. Even more, it has been found an improvement of the interlaminar shear strength of 5% for 3-LED specimens and 10% for 6-LED specimens compared to the specimens cured with UV bulb lamp.

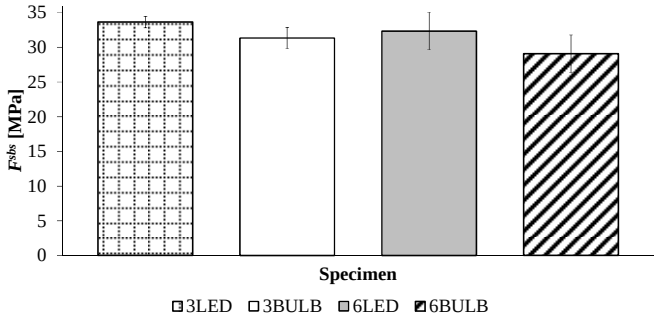


Figure 9. Short-beam strength for manufactured specimens.

4. Conclusions

In the present paper the comparison of the interlaminar properties of a glass-fibre/polyester composite cured with UV LED and conventional flood UV lamp light sources has been carried out. The following conclusions have been obtained:

- The UV LED sources permit a faster curing process than the UV bulb lamps in the same curing conditions due to the higher penetration capacity of its emitting spectrum.
- It can be concluded that the use of UV LED sources for the photopolymerization of thick composites presents an improvement of the curing time.
- Composites cured by UV LED sources present higher interlaminar properties comparing with UV bulb lamp cured composites. In fact, it has been found an improvement of the interlaminar shear strength of 5-10% using the UV LED sources, being higher differences as the thickness of the sample increases.

Acknowledgments

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