



## THE INFLUENCE OF PHOTOPOLYMERIZATION TIME ON THE SORPTION AND SOLUBILITY OF COMPOSITE RESIN

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### ARTICLE INFO

#### Article History:

Received 29<sup>th</sup> June, 2017  
Received in revised form  
22<sup>nd</sup> July, 2017  
Accepted 09<sup>th</sup> August, 2017  
Published online 29<sup>th</sup> September, 2017

#### Keywords:

Photopolymerization,  
Composite Resin,  
Sorption,  
Solubility.

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Citation: Luana Louise Goulart, Priscilla Do Monte Ribeiro Busato, Veridiana Camilotti and Julio Katuhide Ueda. 2017. "The influence of photopolymerization time on the sorption and solubility of composite resin", *International Journal of Development Research*, 7, (09), 15119-15121.

### ABSTRACT

The aim of the study was to evaluate the influence of photopolymerization time of composite resin on the sorption and solubility properties of the material. It was used the Filtek, Z350 XT resin, shade A2B, the study was evaluated in four different timing of light curing (10s, 20s, 40s and 60s). Five resin discs were made per group, of 8mm x 1mm and prepared in accordance with ISO 4049. The water sorption and solubility were calculated using the formulas according to ISO 4049. The results were submitted to analysis of variance (ANOVA) where the time of 40s and 60s polymerization showed the lowest sorption values. The solubility showed no significant differences. With our data it can be concluded that 40s polymerization time must be observed to maintain the properties of the resin.

## INTRODUCTION

A high conversion of the double bonds of carbon into simple results in a resin of better mechanical properties, in terms of: hardness, modulus of elasticity, resistance to fracture, tensile strength and wear resistance. However, such mechanical properties depend on the type of polymer chain and cross-linking density formed during the polymerization process Yap et al. (2004). When a solvent enters the polymer network, it causes an expansion of the structure, facilitating the extraction of unreacted monomers and promotes the dissolution of the linear polymer chains Ferracane JL (2006). This expansion is facilitated when the density of cross-links is low. Thus, cross-linked high density polymers are more resistant to degradation due to greater space limitation and possible pathways for diffusion of the permeants in the polymer structure Ferracane JL (2006). The more cross-linked a polymer is, the lower its sorption and its solubility, which makes it the polymer with better properties Yap (2004), Witzel et al (2005) and Ferracane JL (2006).

In this context, the photopolymerization time and the apparatus that promotes this polymerization are essential for the success of the adhesive restorations. LED light sources appear to be a promising technology for polymerization of resinous materials. Thus, the objective of this study was to evaluate the influence of the LED X light source light curing time on the sorption and solubility properties of the material stored in an ethanol / water solution (75% / 25%). The tested hypothesis was that the polymerization time did not influence the sorption and solubility of the material. The polymerization times investigated were 10s, 20s, 40s and 60s.

## MATERIALS AND METHODS

In order to accomplish this work, twenty composite resin discs (Z350, color A2B, 3M ESPE) were made with a metal structure of 8 mm in diameter and 1 mm in thickness were prepared according to ISO 4049. The specimens were divided into 4 groups (n = 5) according to the polymerization time of 10s, 20s, 40s and 60s, using a light-curing unit (Poly Wireless,

Kavo, Joinville, SC, Brazil), with its tip in contact with the glass coverslip, in order to achieve maximum irradiation.

- Group 1 (n = 5): photopolymerization time of 10s;
- Group 2 (n = 5): photopolymerization time of 20s;
- Group 3 (n = 5): photopolymerization time of 40s;
- Group 4 (n = 5): photopolymerization time of 60s;

The material was inserted into the metal matrix using a syringe (Centrix, DFL Industry and Commerce BC, Rio de Janeiro, RJ, Brazil). A polyester strip and a glass slide were positioned on the inserted composite and the photopolymerized composite using a LED light unit with the light tip in contact with the glass slide to standardize the polymerization distance. The polymerization time was according to each group and the photopolymerizer had its energy density monitored during the experimental procedures using a radiometer. Soon after the samples were prepared according to ISO 4049 specification, the specimens were stored in a desiccator with silica gel at  $37 \pm 1 \text{ }^\circ\text{C}$ . After 24h, the specimens were removed and stored in a desiccator, kept at  $23 \pm 1 \text{ }^\circ\text{C}$  for 1h and then weighed with an analytical balance of 0.2mg accuracy. This desiccation cycle, always using new silica gel, was repeated until a constant mass (m1) was obtained. When the initial desiccation procedure was completed, the specimens were immersed in 1.5 ml of a 75% alcohol / 25% water solution for 7 days at  $37^\circ\text{C}$ . After storage for 7 days, they were removed, washed with water and the excess moisture removed. One minute after removal of the solution were weighed and this mass recorded as m2.

Finally, the specimens were reconditioned to the constant mass, again following the desiccation procedure mentioned above. This constant mass was recorded as m3. The diameter of each specimen and its thickness, measured from the center at four equidistant points, was measured by a precision caliper of 0.01 mm. Then the volume V was calculated in mm<sup>3</sup> according to the equation  $V = \pi (d / 2)^2 h$ , where d is the diameter and h is the thickness of the specimen. The water absorption (Wsp) was calculated in  $\mu\text{g} / \text{mm}^3$  for each of the specimens using the equation  $Wsp = (m2 - m3) / V$ , where m2 is the mass of the specimen after immersion in the alcohol / water solution after 7 days, M3 the mass of the reconditioned specimen and V the volume of the specimen. The solubility in water (Wsl) was calculated in  $\mu\text{g} / \text{mm}^3$  for each of the specimens using the equation  $Wsl = (m1 - m3) / V$ , where m1 is the mass of the specimen conditioned before immersion in water, m3 the mass of Refurbished specimen and V the specimen volume.

After obtaining the results, the data were statically analyzed by ANOVA, at a significance level of 5%, in order to test the effect of the groups.

## RESULTS AND DISCUSSION

The means and standard deviations of the sorption and solubility values can be seen, respectively (Tables 1 and 2). The longevity and clinical success of the restorations are directly dependent on adequate polymerization. Studies have shown that there is a direct relationship between the resistance of the restoration and the efficiency of the polymerization process. We know that the polymerization of photoactivated composite resins occurs in two steps.

**Table 1. Mean values and standard deviation of water sorption**

Group	Wsp ( $\mu\text{g}/\text{mm}^3$ ) (mean $\pm$ SD)
1- Photopolymerization for 10s	18.01 $\pm$ 1.66 <sup>a</sup>
2- Photopolymerization for 20s	17.84 $\pm$ 4.86 <sup>a</sup>
3- Photopolymerization for 40s	11.03 $\pm$ 2.28 <sup>b</sup>
4- Photopolymerization for 60s	10.71 $\pm$ 0.52 <sup>b</sup>

Different lowercase letters mean statistically significant differences ( $p < 0.05$ )

**Table 2. Mean values and standard deviation of water solubility**

Group	Wsl ( $\mu\text{g}/\text{mm}^3$ ) (mean $\pm$ SD)
1- Photopolymerization for 10s	6.67 $\pm$ 7.89
2- Photopolymerization for 20s	4.48 $\pm$ 6.88
3- Photopolymerization for 40s	-1.14 $\pm$ 3.81
4- Photopolymerization for 60s	-1.49 $\pm$ 2.90

There were no statistically significant differences ( $p < 0.05$ )

The first one begins when the material is irradiated by blue light, which stimulates the camphorquinone to react with the tertiary amines and form the free radicals that initiate the chain reaction polymerization by addition. Upon completion of the irradiation, there are still formed free radicals, which continue the polymerization process for about 24 hours. This stage is called post cure 1. Curing effectiveness usually expands with increased exposure to light. This happens because a larger number of free radicals will be formed due to the excitation of the camphorquinone, which directly increases the union of the monomers and the formation of the long molecular chains. By means of a literature review, comparative levels were established between the photopolymerized resins at different times, which evidences evidencing the influence that time had on each group, regarding the characteristics of sorption and solubility.

Sorption and solubility are indirect measures of the degree of polymer polymerization, and are related to the chemistry and structure of these polymer networks, which will determine the extension to which the material will be affected by the surrounding aqueous medium Ferracane JL (2006). Important chemical characteristics include hydrophilicity of the polymer and differences in solubility parameters between polymer and solvent Castro *et al.* (2013). Structural parameters include the density and porosity of the crosslinking network. These properties may also be influenced by the amount and type of inorganic particles present in the material Ferracane JL (2006). According to the present results, the sorption showed statistically significant differences between the groups, in which a longer polymerization time (40s and 60s) led to a lower incorporation of the solvent in the composite. Composites activated for a longer time generally exhibit a higher degree of conversion of monomers to polymers Castro *et al.* (2013) and, consequently, a higher degree of interlacing of the linear chains, leading to lower values of sorption Castro *et al.* (2013). This is because a larger number of free radicals will be formed due to the excitation of the camphorquinone, which directly increases the union of the monomers and the formation of the long molecular chains Albuquerque *et al.* (2013). This higher degree of conversion forms less hydrophilic sites in the material, in other words, a lower chemical affinity of the polymer with the solvent (there are fewer unreacted monomers) and also less space available for solvent penetration between the polymer chains (More crosslinking), leading to lower sorption Castro *et al.* (2013). The experiment showed that the resins exposed to a longer time of photopolymerization showed lower sorption of water.

In the oral cavity this is extremely important, since it is always moist due to the saliva. Therefore, the dental surgeon must expose the resin to a time between 40s and 60s of photopolymerization so that the restoration has a greater durability, as sorption of liquids will deteriorate the resin over time Ferracane JL (2006). Regarding solubility there were no statistically significant differences, despite its increase with the decrease of the polymerization time. Even so, for solubility, a polymerization time of 40 seconds would be required to provide better properties for the resin evaluated. A similar result was found in the literature Castro *et al* (2013) and explanations for this fact are due to factors such as the short storage period in the solvent, which was 7 days, and may not have been sufficient to show any existing differences. Each resin has a distinct period for liquid absorption, so that this short time of alcohol / water storage may have been insufficient to modify the properties of the investigated material. Perhaps longer times where the solvent could cross the surface layer of the more polymerized resin are needed to find differences.

### Conclusion

It was concluded that the polymerization time of 40s should be respected for the maintenance of the resin properties, that is, the adequate photopolymerization time means quality and durability of the restoration.

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