Degree of conversion of 3D printing resins used for splints and orthodontic appliances under different post-polymerization conditions.

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Research Article

Keywords:

Posted Date: June 2nd, 2022

DOI: https://doi.org/10.21203/rs.3.rs-1661000/v1

Additional Declarations: No competing interests reported.

Version of Record: A version of this preprint was published at Clinical Oral Investigations on February 9th, 2023. See the published version at https://doi.org/10.1007/s00784-023-04893-8.

Abstract

Objectives: To measure the degree of conversion (DC) of different 3D printing resins usable for splints or orthodontic appliances, manufacturing under different post-polymerization conditions.

Materials and Methods: Five 3D printing photopolymer resins were studied. Each resin was analyzed in liquid form (n=15), then cylindrical specimens (n=135) were additively manufactured and post-cured with different times (10, 60 and 90 minutes) and temperatures (20°, 60° and 80°). The degree of conversion of each specimen was measured by Fourier Transformed Infrared Spectroscopy (FTIR). The data were statistically analyzed using a 3-way ANOVA followed by Tukey's post hoc test.

Results: Time and temperature of post-polymerization significantly influenced the DC of each resin: when time and/or temperature increased, the DC increased. For all resins tested, the lowest DC was obtained with a post-curing protocol at 10 min and 20° and the highest DC at 90 min 80°. However, at 80°, samples showed a yellowish color.

Conclusions: The time and temperature of post-curing could have an impact on the DC of the 3D printing resins studied. DC of the 3D printing resins could be optimized by adjusting the post-polymerization protocol.

Clinical Relevance: Whatever the resin used, it seems that a post-curing at 60 min and 60° would be the minimal time and temperature conditions to obtain a proper polymerization. Beyond that, to boost the DC, it would be preferable to increase the post-curing time

Introduction

The recent development of additive manufacturing, also known as 3D printing, in dentistry opens new perspectives [1, 2]. Technologies mainly used include stereolithography (SLA) and Digital light processing (DLP) each one offering advantages depending on the type of object which is created [2, 3]. These techniques polymerize liquid photosensitive resins to build up an object with a laser beam (SLA) or a light beam (DLP). After printing, object is usually cleaned with 99% isopropylic alcohol (IPA) to remove uncured resins and then post-cured in a light-curing unit (LCU), as recommended by manufacturers to improve mechanical properties and complete the polymerization process [3–6].

In orthodontics, fabrication of occlusal splints, retainers and clear aligners usually involve thermoforming of different thermoplastic sheets on models. The latter can be plaster models or currently 3D printed models [7, 8]. Ideally, 3D printing resins should be used to make directly occlusal splints, retainers, and clear aligners to bypass model printing and thermoforming process, increasing efficiency, and decreasing the environmental impact [1, 7, 9–11].

However, while thermoplastic materials are very biocompatible [8, 12–16], few studies have looked at the biocompatibility of 3D printing resins indicated for intraoral use [11, 12]. These resins are generally photopolymers composed of methacrylate, also found in the composition of bonding materials used in conventional fixed technique [3, 11, 12]. Thus, they could release monomers into the oral environment. In addition, unlike brackets bonded to buccal or lingual of each tooth where the release of monomers takes place on a very small area (corresponding to the bonding joint and a slight area of excess on the surface of the tooth), an occlusal splint or a clear aligner cover the entire surface of all teeth. The release of monomers could be much more important [17].

The degree of conversion (DC) of a material gives a first idea of its biocompatibility [18, 19]. It evaluates the conversion rate of monomers into polymers during the polymerization and therefore the rate of residual monomers likely to be released. DC also plays an important role in mechanical properties or color stability [18–23]. For an object fabricated with SLA or DLP process, DC depends on several factors such as print layer thickness and post-curing conditions (time, temperature, intensity of light source etc) [21–28]. One method for measuring the DC of a material is the Fourier Transformed Infrared Spectroscopy (FTIR) which determine vibrations of C = C double bonds involved in polymerization process [18, 19]. Contrary to orthodontic bonding materials [29–33], there are no studies on the DC of 3D printing resin used for orthodontic appliances.

The aim of this study was to measure the DC of five 3D printing resins usable for occlusal splints, retainers and potentially clear aligners, manufactured under different post-polymerization conditions.

Materials And Methods

Materials tested

Five resins for 3D printing of orthodontic splints were tested (Table 1):

- Dental LT (V2) (Formlabs)
- Ortho Clear (NextDent)
- Ortho Rigid (NextDent)
- Ortho IBT (NextDent)
- Keysplint soft (Keystone industries)

Table 1
Materials, manufacturers, batch number and chemical composition

Materials		Manufacturer	Batch number	Composition	Instructions for use IPA, time and T° of post-curing
3D resins	**Dental LT Clear (V2)**	Formlabs, Somerville, MA, USA	DC0120061101	UDMA (50–75%) HEMA (10–20%) EGDMA (< 10%) HPA (0,1–1,5%)	IPA: 20min Time: 60° (full cure) T°: 60° (full cure)
	Ortho Clear	NextDent, Soesterberg, Netherlands	XG393N03	Methacrylates oligomers	IPA: 5min Time: 10min T°: not specified
	Ortho Rigid	NextDent, Soesterberg, Netherlands	WY153N030	Methacrylates oligomers	IPA : 5min Time: 10min T°: not specified
	Ortho IBT	NextDent, Soesterberg, Netherlands	XG283N01	Methacrylates oligomers	IPA : 5min Time: 10min T°: not specified
	Keysplint Soft	Keystone industries, Myerstown, PA, USA	KI0410	Methacrylates Photo-initiator, inhibitor and pigment	IPA : 5 min Time: 20 min T°: not specified

For specimen fabrication, a cylinder of 30mm of diameter and 2mm of thickness was designed with CAD software Fusion 360® (Autodesk, San Rafael, USA) and data export in STL (Standard Tessellation Language) format.

Samples preparation and protocols tested

First, each resin studied was analyzed before printing in liquid, unpolymerized, state (3 measures on 3 specimens, 5 x n = 3x3). Then, each resin was analyzed in polymerized state, after printing and post-processing (3 measures on 3 specimens, in 9 post-curing protocols 5 x 9 x n = 3x3).

Two printers were used for specimens manufacturing: a DLP printer (Sprintray Pro 95, Sprintray, USA) for Ortho Clear, Ortho Rigid, Ortho IBT and Keysplint soft resins and a SLA printer (Form 2, Formlabs, USA) for Dental LT resin. For each resin, printing was carried out with a layer thickness of 100µm, an angulation of 45° and the addition of supports for a better adhesion of pieces on printer tray.

After printing, specimens were cleaned with isopropylic alcohol (IPA) during 20 minutes for Dental LT and 5 minutes for other resins according to manufacturer's recommendation's for removing the excess of uncured resin (FormWash, Formlabs, USA). Then, they were all post-cured with the same Light Curing Unit (Form Cure, Formlabs, USA) with 405nm wavelenght according different protocols: 3 different times (10, 60 and 90 min) and 3 different temperatures (20°, 60° and 80°) (Table 2).

Table 2
Different post-processing protocols applied for each resin

Materials	IPA Post-rinsing	Time of post-curing	Temperature of post-curing
Dental LT	20 min	10 min, 60 min,90 min	20°, 60°, 80°
Ortho Clear	5 min		
Ortho Rigid	5 min		
Ortho IBT	5 min		
Keysplint Soft	5 min		

After post-processing, supports were removed with a resin bur and specimens polished with sandpaper (800 grits).

Measure of degree of conversion (DC)

Degree of conversion was measured with Fourier Transformed Infrared Spectroscopy (FTIR) with Nicolet™ iS10 spectrometer (ThermoFischer Scientific) in ATR (Attenuated Total Reflectance) mode. Spectra were recorded (Fig. 1) with OMNIC software (Thermo Electron Corporation).

After background, all measurements were obtained under the following parameters: resolution of 4cm^{-1} and 32 internal scans per reading. For each specimen, spectra were recorded 3 times.

Degree of conversion was determined with the evaluation of change in height ratio of the absorbance of the aliphatic C = C peak (1638 cm^{-1}) and aromatic C = C peak (1608 cm^{-1}) during polymerization process. The following formula was used:

$$DC\,(\%) = \left(1 - \frac{Abs\frac{C=C1638cm^{-1}(P)}{C=C1608cm^{-1}(P)}}{Abs\frac{C=C1638cm^{-1}(NP)}{C=C1608cm^{-1}(NP)}}\right) X100$$

Abs: Absorbance

P: polymerized

NP: non polymerized

For Dental LT and Keysplint soft, peaks corresponding to C = C aromatic double bonds at 1608 cm^{-1} were replaced by peaks of C = O double bonds at 1720 cm^{-1}. This peak is also a reference peak, stable and easy to identify in spectra.

Statistical analysis

DC was expressed in percent by means and standard deviations. Normal distribution was confirmed by a Shapiro-Wilk test and the equality of variances assessed with a Levene test. A 3-way-ANOVA followed by Tukey's post hoc test was performed for the factors "resin", "time" and "temperature". For all the tests, the significance level was set at $p < 0.05$. Tests were carried out with XLSTAT (Addinsoft, Paris, France) and Graphpad Prism 9 software (Graphpad Software Inc, California, USA).

Results

DC values obtained for each 3D printing resin are reported in Table 3 and summarized in Fig. 2.

Table 3
Means and standard deviations of DC (in %) obtained with each resins according to the time and temperature of post-curing

Time	**10 min**			**60 min**			**90 min**		
Temperature	**20°**	**60°**	**80°**	**20°**	**60°**	**80°**	**20°**	**60°**	**80°**
Dental LT	54.6^{nopq} ± 1.9	67.6^{ijkl} ± 1.7	74.0^{defgh} ± 1.6	61.0^{klmn} ± 2.0	81.6^{abc} ± 1.0	$83 .0^{ab}$ ± 0.8	62.0^{jklm} ± 1.8	83.5^{ab} ± 1.0	85.4^{a} ± 0.7
Ortho Clear	41.0^{t} ± 2.2	54.8^{mnopq} ± 3.4	67.0^{hijk} ± 3.5	52.7^{opq} ± 5.3	74.5^{cdefg} ± 2.2	80.6^{abcde} ± 2.4	57.6^{lmnop} ± 3.2	81.4^{abc} ± 2.4	82.7^{ab} ± 1.8
Ortho Rigid	37.8^{t} ± 2.5	51.9^{pq} ± 2.6	60.9^{klmn} ± 2.5	40.9^{t} ± 0.5	69.6^{ghi} ± 1.3	73.9^{efgh} ± 1.8	44.5^{rst} ± 2.4	73.5^{efgh} ± 2.5	75.2^{cdefg} ± 2.5
Ortho IBT	40.5^{t} ± 3.1	56.5^{mnop} ± 3.3	68.1^{ghijk} ± 2.3	42.8st ± 1.9	73.0^{fgh} ± 2.4	78.3^{abcdef} ± 1.7	50.5^{pqr} ± 2.7	77.9^{bcdef} ± 1.2	80.5^{abcde} ± 2.2
Keysplint Soft	40.3^{t} ± 2.5	59.4^{lmno} ± 2.3	68.5^{ghij} ± 2.2	49.2^{qrs} ± 3.9	78.4^{abcdef} ± 2.4	81.1^{abcd} ± 2.7	51.3^{pqr} ± 2.7	81.0^{abcd} ± 1.9	83.1^{ab} ± 1.2
Values with the same letter in exponent are not significantly different at p < 0.05									

Time and temperature of post-polymerization significantly influenced the DC of each resin: when time and/or temperature increased, the DC increased. For all resins tested, the lowest DC was obtained with a post-curing protocol at 10 min and 20° (54.6 ± 1.9 for Dental LT, 41.0 ± 2.2 for Ortho Clear, 37.8 ± 2.5 for Ortho Rigid, 40.5 ± 3.1 for Ortho IBT and 40.3 ± 2.5 for Keysplint soft) and the highest DC at 90 min 80° (85.4 ± 0.7 for Dental LT, 82.69 ± 1.8 for Ortho Clear, 75.16 ± 2.5 for Ortho Rigid 80.52 ± 2.2 for Ortho IBT, and 83.15 ± 1.2 for Keysplint soft).

For a time of 10 min, the temperature significantly influenced the DC between 20° and 60°, but also between 60° and 80°. For a time higher than 60 min, the temperature significantly influenced the DC between 20° and 60°, but not between 60° and 80°.

For a temperature of 20°, the time significantly influenced the DC between 10 and 60 min, but also between 60 and 90 min. For temperatures higher than 60°, the time generally significantly influenced the DC between 10 and 60 min, but not between 60 and 80 min.

Besides, even if the DC values of Dental LT resin were significantly higher than those of other resins studied for some time and temperature, there was no significant influence of the type of resin on DC ($p > 0,05$).

At last, at 80°, samples showed a yellowish color

Discussion

Effect of time and temperature on DC

Results of this study showed that DC of 3D printing resins significantly increased with post-polymerization time and temperature. These results are confirmed by the findings of other studies [23–27]. Indeed, when temperature increases, viscosity of resin decreases, thus frequency of collisions between macromolecular chains increases and energy required to initiate polymerization reactions decreases, making monomer polymerization easier [23]. In addition, the number of residual monomers decreases with a longer polymerization time [23, 25, 26]. However, the use of high post-curing temperatures could affect material properties if not properly controlled. Some authors have reported this would hasten the aging mechanism of the material [22–28].

DC values for each resin were higher compared to DC values of adhesive and resin composites [29–33]. 3D printing resins are composed of methacrylate like resin composites used for bonding brackets but contain less or no fillers in their matrix, which could explain these higher DC values [3, 21, 22]. Indeed, it has been reported that a direct resin composite has a lower DC if fillers rate in its matrix is high [29–33]. Differences in DC values could also be explained by the implementation technique. With SLA or DLP, material is polymerized in thinner layers than a material used in direct technique [2, 4, 6]. And, with 3D printing, the post-curing protocol applied to the material increases its DC [6, 23–26].

Nevertheless, DC values of 3D printing resins are not of 100%. In conventional technique with brackets, the surface exposed of resin composite is limited to excess and joint and with an occlusal splint or a clear aligner, the surface developed covers the entire surface of each tooth [17]. However, materials for thermoforming stay in polymer state, without leading monomer release above the toxicity rate. [12–16]. So, 3D printed occlusal splints or clear aligners could release more monomers than previous techniques.

Effect of 3D resin and other parameters on DC

Our study showed no significant influence of the type of resin on DC. Each resin differs by its chemical composition (in particular photo-initiators concentration) and by their polymerization kinetics [3, 21]. A study showed that DC increases with the concentration of photo-initiators when concentration remains low but decreases at high concentration of photo-initiators [33]. Moreover, printing technique used depends on resins studied. Dental LT is a resin printed by SLA while the four other resins are printed by DLP. Some authors showed an influence of printing technique on DC particularly on printing parameters (print layer thickness, deposition time of each layer…) [25–28]. A study reported that print layer thickness would influence DC: a layer thickness of 50 µm or 100 µm gives better DC than 25 µm [25]. In our study, we used the same print layer thickness for each resin (100 µm).

Type of equipment used for post-curing (Light curing unit or LCU) would also influence DC, due to different parameters: including type of the light source (UV or LED), light intensity [26], spectrum of absorbed wavelengths, irradiation type (constant or with repeated flashes) [3, 23, 25, 26], time and temperature. The position of the specimen in the device has also an impact on DC [25, 26]. Normally, each manufacturer recommends post-curing their resins with their specific LCU. However, in our study, we post-cured all specimens with the same LCU (Form Cure at a wavelength of 405 nm) in order to study the mechanism of polymerization. That would explain the slight best DC values for the Dental LT resin. Besides, it shows that all devices can be used for all resins in adapting the LCU parameters. Moreover, shorter post-curing time are recommended for the other resins, because their specific LCU has highest irradiance than Form Cure [26].

Comparison with manufacturer recommendation

For Dental LT, manufacturer defines two post-curing protocols: a classic protocol of 30 min at 60° and an optimized protocol (full post-cure) of 60 min at 60°. Our results showed a higher DC value for a longer time (83,5%) or a higher temperature (83% at 60 min 80° and 85,4% at 90 min 80°), however without significant differences. So, the full post-cure post-polymerization protocol recommended by the manufactured would allow an optimal DC value.

For Ortho IBT, Ortho Rigid and Ortho Clear, manufacturer recommends a 10 min post-curing protocol without specifying any associated temperature. Likewise, for Keysplint soft, manufacturer indicates a 25 min post-curing protocol without associated temperature. (These shorter times are probably related to their specific LCU devices with higher irradiance than Form Cure used in this study [3, 6]). For these four resins, when a time of 10 min is chosen, a temperature of 80° allows a significant increase of DC, but without reaching the highest DC. Therefore, an increase of time such as 60 min, is necessary. On the contrary, at a temperature of 20°, a time of 60 min and even 90 min did not allow or almost an increase of DC. Consequently, increasing temperature such as 60°, is necessary. However, with the specific LCU device for these four resins, the control of the temperature is unknown [3, 26].

Thus, whatever the resin used it seems that a post-curing at 60 min and 60° would be the minimal conditions to obtain a proper polymerization. Then, an increase of temperature to 80° or time to 90 min can significantly often improve the DC values. Besides, at 80°, samples showed a yellowish color. This could be explained by a competition between photo and thermo-polymerization processes. When thermo-polymerization is too quick, free radicals of an initiator/co-initiator couple (probably phosphonyl emitted by the phosphine oxides such as BAPO) are unable to polymerize because blocked in the resin already cured, giving this yellow color [3, 21]. For this reason, it would be preferable to increase post-curing time to boost the DC [3, 21, 26].

At last, higher post-curing time and/or temperatures values could affect mechanical properties or microscopic deformations [22–28].

Conclusion

This study showed an impact of post-curing time and temperature on DC of 3D printing resins that could be used in orthodontics for manufacturing occlusal splints, retainers or clear aligners.

In our study, it seems that a minimum time (around 60 min) combined with a minimum temperature (around 60°) would give a satisfactory DC. While a higher temperature increases the DC but cause a yellowish resin coloration, a longer time could contribute to improve the DC without color changes. Likewise, it would be relevant to assess if a longer time and/or a higher temperature could affect their mechanical properties.

Further studies on the release of monomers are needed to complete the DC data regarding the biocompatibility of these resins.

Declarations

Acknowledgments:

To Hélène Gouze for her valuable help in the statistical analysis.

CONFLICT OF INTERESTS

The authors declare any financial and non-financial conflict of interests.

ETHICS APPROVAL AND CONSENT TO PARTICIPATE

Not Applicable

FUNDING

No funding was obtained for this study.

AUTHOR CONTRIBUTION

ED designed the study.

CA did the experiment, helped by PF and SLG.

CA, ED and PF wrote the manuscript.

All authors reviewed the manuscript.

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Figures

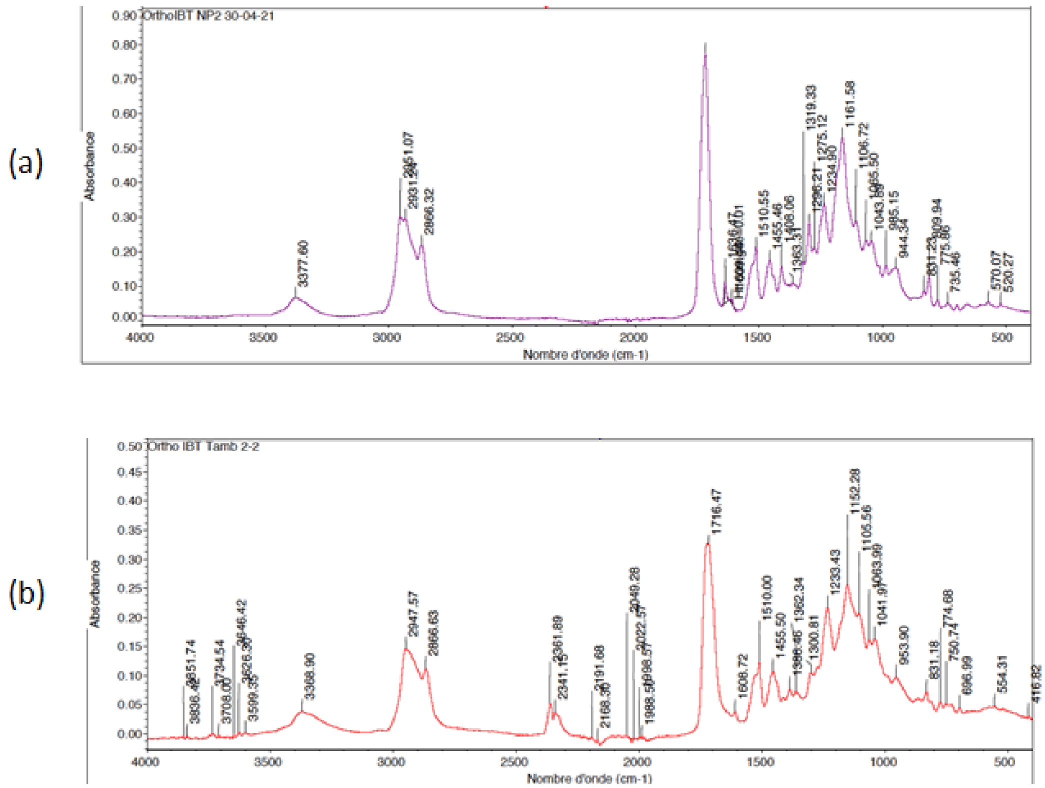
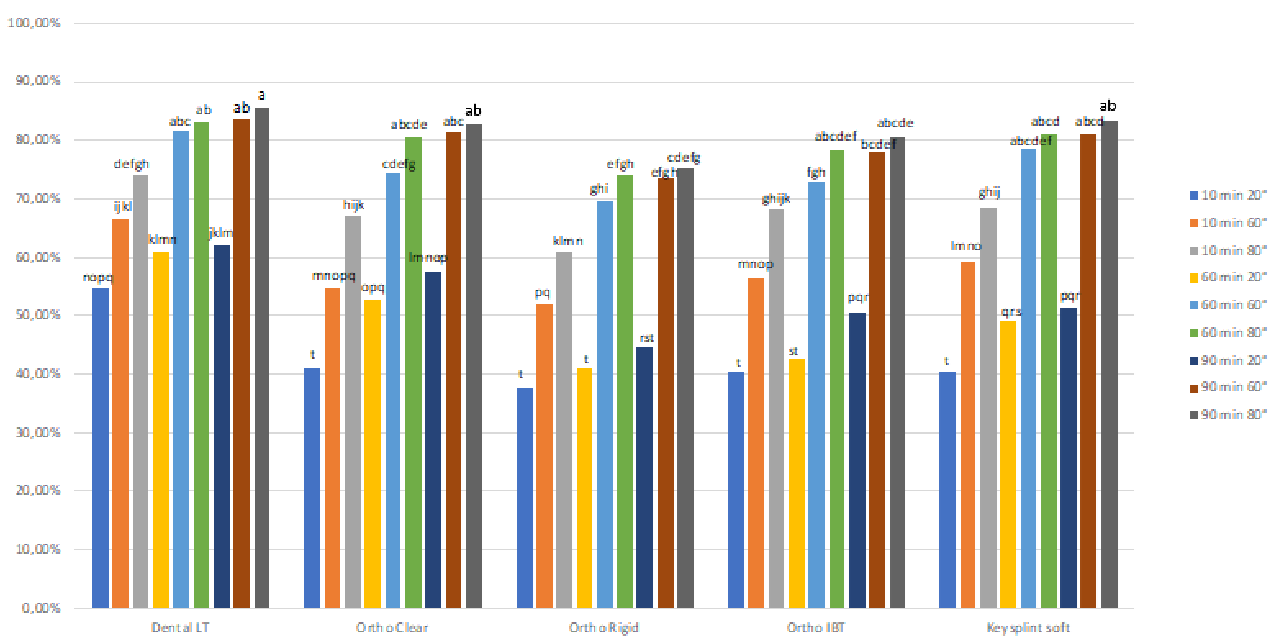


Figure 1

a. Example of spectra recorded for Ortho IBT® resin at unpolymerized state.

b. Example of spectra recorded for Ortho IBT® resin at polymerized state (10 min 20°).



Values with the same letter in exponent are not significantly different at p<0,05

Figure 2

Comparison of DC of each post-curing protocol for each resin.