Specac

Application Note

Study of Polymerization Kinetics of Dental Materials using the Golden Gate® ATR

Golden Gate ATR

INSIDE: Investigate chemical composition of dental bonding systems and to assess their polymerization kinetics upon light exposure for 20 seconds

Introduction

Dental composites are a suspension of inorganic glass particles in a polymerizable organic matrix of monomethacrylate and dimethacrylate monomers such as Bis-GMA, TEGDMA, UDMA and HEMA [1]. The bonding systems are made up of resin monomers and may contain fillers. These are generally hydrophilic/hydrophobic, solvated monomeric mixtures which may come in separate bottles or together in a single bottle [2]. Such materials polymerize via a free-radical polymerization reaction, where a vinyl C=C bond is broken into a single bond [3]. This reaction is generally initiated by visible blue light, as they have a photoinitiation system within their composition. To characterize these materials, ATR-FTIR techniques are commonly used and are preferred over alternatives [4-7].

Acknowledgement

António HS Delgado[1,2], Anne Young[1]

[1]Division of Biomaterials and Tissue Engineering, UCL Eastman Dental Institute, Royal Free Hospital, London, UK

[2]Centro de Investigação Interdisciplinar Egas Moniz (CiiEM), Instituto Universitário Egas Moniz, Almada, Portugal

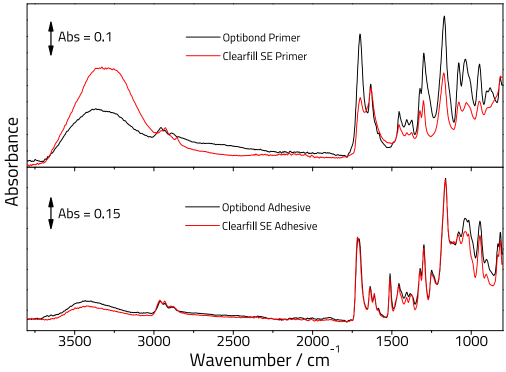

Figure 1: ***FTIR spectra of primers (top) and adhesives (bottom).***

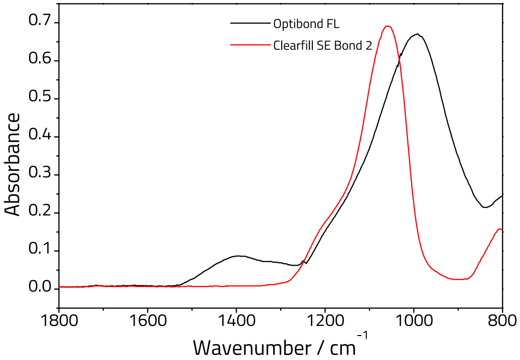

Figure 2: ***FTIR spectra of the fillers.***

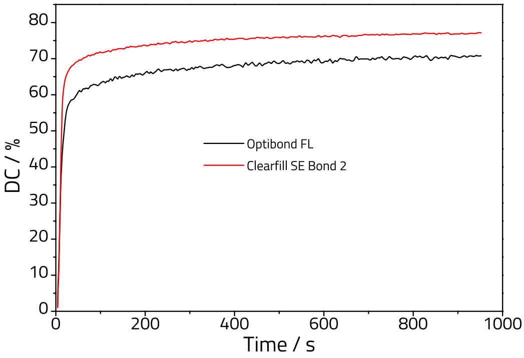

Figure 3: ***Degree of conversion (D_c) as measured from the height of the methacrylate peak at 1320 cm^{-1} for the two bonding systems.***

Experimental

Two commercial dental bonding systems were tested, Optibond FL (Kerr, Orange, CA, USA) and Clearfill SE Bond 2 (Kuraray, Tokyo, Japan). A Golden Gate diamond ATR accessory coupled with a commercially available FTIR spectrometer was used to collect spectra of separate primers/adhesives. Spectra of isolated fillers were obtained following their separation from the adhesives, and filler loads by analyzing three repetitions using a gravimetric method (n=3). The bridge of the Golden Gate ATR system was pressed down to ensure contact of the diamond with the solid fillers. Spectral changes during light exposure (20 s, LED curing unit 1100–1330 mW cm^{-2}) were used to determine polymerization kinetics (n=3), by recording time-resolved spectra before, during, and after light exposure, for 1000 s.

Results and Discussion

The spectra of primers and adhesives (Fig. 1) are dominated by solvent peaks (3300 cm^{-1} and 1635 cm^{-1}), with Clearfill SE Bond 2 having double the solvent content of Optibond FL. Adhesives show similar spectra, compatible with the presence of hydrophobic monomers, with Bis-GMA C=C aromatic peaks at 1610 cm^{-1} and 1510 cm^{-1}.

FTIR spectra of the two fillers are shown in Figure 2. They revealed a strong, broad band at 992 cm^{-1} resulting from overlapping contributions of Si-O bond stretching and B-O vibration in the barium aluminoborosilicate glass. Optibond FL has a peak in the 1400 cm^{-1} region due to B-O stretching. Si-CH2 in silanated particles is seen at 1200 cm^{-1}, while the peak at 1060-1080 cm^{-1} is due to Si-O asymmetric stretch of silica particles. The 790 cm^{-1} peak is due to symmetric stretching of Si-O. The percentage degree of conversion (D_c) is calculated using 100 x $(h_0-h_t)/h_0$, where h_0 and h_t represent the height of the reaction methacrylate peak (1320 cm^{-1}) above background, initially and at time t, after start of the polymerization. Light curing began 20 s after start of spectral acquisition and lasted for 20 s. Bonding systems reach or surpass 50% degree of conversion D_c (%) before light irradiation stops. Clearfill SE Bond 2 attains higher final D_c (%) compared to Optibond FL (Fig. 3).

Conclusion

The ATR-FTIR method is very useful to characterize the chemistry of dental bonding systems, as a monomeric liquid mixture but also of the separated solids, in this case, the inorganic glass fillers. Continuous spectral acquisition during photopolymerization, without any disconnect from the ATR, allows monitoring of the polymerization reaction and assessment of the degree of conversion D_c (%), with high reproducibility and without the need for normalization.

References

[1] Aminoroaya A, Neisiany RE, Khorasani SN, Panahi P, Das O, Ramakrishna S, ***ACS Biomater. Sci. Eng.***, **6**, (2020), 3713–44. DOI: 10.1021/acsbiomaterials.0c00051

[2] Meerbeek BV, Yoshihara K, Landuyt KV, Yoshida Y, Peumans M, ***J. Adhesive Dent.***, **22**, (2020), 7–34. DOI: 10.3290/j.jad.a43994

[3] Moszner N, Hirt T, ***J. Polym. Sci. Part Polym. Chem.***, **50**, (2012), 4369–402. DOI: 10.1002/pola.26260

[4] Wang Y, Spencer P, Yao X, Brenda B, ***J. Biomed. Mater. Res. - Part A***, 82, (2007), 975–83. DOI: 10.1002/jbm.a.31232

[5] Amirouche-Korichi A, Mouzali M, Watts DC, ***Dent. Mater.***, **25**, 2009, 1411–8. DOI: 10.1016/j.dental.2009.06.009

[6] Okulus Z, Buchwald T, Szybowicz M, Voelkel A, ***Mater. Chem. Phys.***, **145**, (2014), 304–12. DOI: 10.1016/j.matchemphys.2014.02.012

[7] Delgado AH, Young AM, ***Materials***, **14**, (2021), 760. DOI: 10.3390/ma14040760

AN21-06

United Kingdom
sales@specac.co.uk

China
frank.li@specac.com

United States
sales@specac.com

Singapore
kamhar.woo@specac.com

UK: +44 (0)1689 892 902 | US: +1 866 726 1126

Specac brilliant spectroscopy™

www.specac.com

Follow us: