JOURNAL OF THE MECHANICAL BEHAVIOR OF BIOMEDICAL MATERIALS 29 (2014) 508–516

Available online at www.sciencedirect.com

ScienceDirect

www.elsevier.com/locate/jmbbm

Research Paper

Quantitative wear and wear damage analysis of composite resins *in vitro*

Natthavoot Koottathape[a], Hidekazu Takahashi[b,*], Naohiko Iwasaki[b], Masafumi Kanehira[c], Werner J. Finger[d]

[a]Advanced Biomaterials, Department of Restorative Sciences, Division of Oral Health Sciences, Graduate School of Medical and Dental Sciences, Tokyo Medical and Dental University, 1-5-45 Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
[b]Oral Biomaterials Engineering, Department of Oral Materials Sciences and Technology, Course for Oral Health Engineering, School of Oral Health Care Sciences, Faculty of Dentistry, Tokyo Medical and Dental University, 1-5-45, Yushima, Bunkyo-ku, Tokyo 113-8549, Japan
[c]Division of Operative Dentistry, Department of Restorative Dentistry, Tohoku University Graduate School of Dentistry, 4-1 Seiryo-machi, Aoba-ku, Sendai 980-8575, Miyagi, Japan
[d]Liaison Center for Innovative Dentistry, Tohoku University Graduate School of Dentistry, 4-1 Seiryo-machi, Aoba-ku, Sendai 980-8575, Miyagi, Japan

ARTICLE INFO

Article history:
Received 17 September 2013
Accepted 2 October 2013
Available online 20 October 2013

Keywords:
Composite resin
Wear resistance
Volume loss
Wear damage analysis

ABSTRACT

The aim of this study was to investigate volume loss and worn surfaces' morphologies of eight composite resins: Durafill VS (DUR), Clearfil AP-X (APX), Filtek Z250 (Z250), Filtek Supreme XT (FIL), Kalore (KAL), MI Flow (MFL), Venus Diamond (VED) and Venus Pearl (VEP). Disc-shaped specimens were fabricated and mounted in a ball-on-disc wear testing machine and abraded in water or with the third-body media, poppy seed slurry and polymethyl methacrylate (PMMA) slurry. Volume loss ($n=5$) was determined after 50k sliding cycles, and analyzed using two-way ANOVA ($\alpha=0.05$). The worn surfaces were examined with SEM. Two-way ANOVA suggested significant interaction between composite and wear condition. DUR, KAL and MFL showed low wear in water. DUR, Z250 and FIL showed moderate wear with PMMA slurry, whereas APX, KAL and MFL were deeper abraded. Under the action of poppy seed slurry DUR proved high volume loss. SEM showed that Z250, FIL and MFL were uniformly abraded in water. KAL and MFL with poppy seed were heavily destructed, whereas VED and VEP appeared very smooth. KAL and MFL abraded with PMMA slurry showed many cracks, but VEP remained crack-free and smooth. Volume loss and worn surfaces' morphologies varied with type of composite and third-body media used.

* *Corresponding author*. Tel./fax: +81 3 5803 5379.
E-mail addresses: takuabm@tmd.ac.jp (N. Koottathape), takahashi.bmoe@tmd.ac.jp (H. Takahashi), iwasaki.bmoe@tmd.ac.jp (N. Iwasaki), kane@m.tohoku.ac.jp (M. Kanehira), wjfinger@dent.tohoku.ac.jp (W.J. Finger).

1751-6161/$ - see front matter © 2013 Elsevier Ltd. All rights reserved.
http://dx.doi.org/10.1016/j.jmbbm.2013.10.003

1. Introduction

Composite resins have been continuously and successfully developed throughout the last decades. Major improvements were achieved regarding clinical appearance, physical and mechanical properties. However, clinical failures of composite resin restorations are still frequently reported. According to long-term clinical studies, commonly reported failures were secondary caries with a failure rate of 9.1–24.5% and restoration fractures with a failure rate of 12–19% (Brunthaler et al., 2003; Da Rosa Rodolpho et al., 2011; Demarco et al., 2012; Sarrett, 2005; Van Dijken, 2000). Insufficient wear resistance of posterior composite resin restorations was reported with a failure rate of 6.1% (Van Dijken, 2000). It has however to be kept in mind that the resin restorations referred to in the articles above do not represent more recent composite types, such as micro-hybrid, nano-hybrid and nanofilled materials. Controlled clinical studies on posterior restorations using hybrid, micro-hybrid and nanofiller containing composites revealed that there was no significant difference in wear (Frankenberger et al., 2013; Garcia-Godoy et al., 2012; Krämer et al., 2011; Palaniappan et al., 2011; Schirrmeister et al., 2009; Yesil et al., 2008). Hamburger et al. (2011) reported good clinical performance after a mean observation time of 3.9 years of hybrid-type composite restorations that were placed in patients with severe tooth wear requiring an increase of the occlusal vertical dimension. In spite of the considerable progress achieved wear of composite resin is still considered a concern regarding the longevity of posterior restorations, especially with large restorations in molars and patients with bruxing habits (Ferracane, 2006).

Prediction of the expected clinical performance of new composite resins derived from laboratory tests is highly desirable. However, the clinical success of composite resins is multifactorial and unlikely to be predicted accurately *in vitro*, even when using a battery of testing methods (Ferracane, 2013). Therefore, *in vitro* wear testing is primarily useful to categorize composites in terms of product rankings. DeLong et al. (2012) reported a good clinical correlation with artificial mouth data in two-body wear condition; however no third-body medium was used in their study.

Many articles on *in vitro* wear of composite resins have been published during the years, using a variety of different devices to simulate two- and/or three-body wear modes (Condon and Ferracane, 1997; Hahnel et al., 2011; Heintze et al., 2011; Hu et al., 2002; Koottathape et al., 2012b; Lambrechts et al., 1987, 2006; Zantner et al., 2004).

When simulating *in vivo* conditions with *in vitro* test regimens quantitative evaluation of both two-body and three-body wear should be required (Hahnel et al., 2011; Heintze, 2006). Selection of the third-body medium is crucial for simulation of clinical wear (Lawson et al., 2012). The ISO technical specification 14569-2:2001 "Wear by two- and/or three-body contact" (ISO/TS 14569-2, 2001) suggests both natural grains such as poppy seed and synthetic material polymethyl methacrylate (PMMA) as abrasive particles. When the Oregon Health Sciences University oral wear simulator (OHSU) was operated with a mixture of PMMA and poppy seed a reasonably good correlation with clinical wear data was found, whereas other *in vitro* wear tests investigated gave some product ranking only that did not reflect clinical performance (Heintze et al., 2012).

In an attempt to elucidate wear mechanisms it is mandatory to determine quantitative wear parameters as well as the worn surfaces' morphology. The number of wear cycles applied must be large enough to detect any non-linearity in wear, eventually caused by major failure events, typically fatigue failures. As an example, microfilled composites showed low wear and negligible fracture during three or four years of service, before catastrophic failure occurred (Lambrechts et al., 1987).

The aim of the present *in vitro* wear trial was to investigate eight composite resins, thereof three conventional reference materials and five composites including nanofiller particles, regarding volume loss after 10,000, 30,000 and 50,000 sliding cycles in a ball-on-disc sliding device in water or with two different third-body media, and to examine the worn surfaces by scanning electron microscopy in order to detect morphological details, pointing on or supporting hypotheses about the wear mechanisms involved. The null hypotheses tested were that there would be no significant difference in volume loss and no difference in microscopic appearance of the wear pattern morphology among the composite resins investigated.

2. Materials and methods

Table 1 shows the eight composite resins tested and their compositions as publicly available manufacturer information. Three conventional composites were selected as reference materials: Durafill *VS* (DUR; microfilled), Clearfil AP-X (APX; hybrid) and Filtek Z250 (Z250; micro-hybrid). The remaining composite resins contain nanofiller particles: Filtek Supreme XT (FIL; nanofilled), Kalore (KAL; nano-hybrid), MI Flow (MFL; nano-hybrid, flowable), Venus Diamond (VED; nano-hybrid) and Venus Pearl (VEP; nano-hybrid).

2.1. *Specimen preparation*

For each material 15 composite resin specimens were produced in cylindrical aluminum molds (8 mm in diameter, 2 mm in depth). The molds were filled, covered with a Mylar strip and pressed flush with a glass slide prior to 40 s halogen light curing (XL 3000, 3 M ESPE, MN, USA; output $>$ 500 $mWcm^{-2}$) with the light exit window in contact with the covering strip. After one week of storage in 37 °C water composite surplus was carefully removed (grinding and polishing on wet SiC paper, grits #600, 1,500 and 4,000) and two reference points were created on alternate peripheral sites of the polished surface (Diamond Point #40, Shofu, Kyoto, Japan). Following ultrasonic cleaning five specimens from each material group were allocated to test under two- and two different three-body wear conditions.