An approach to understanding tribological behaviour of dental composites through volumetric wear loss and wear mechanism determination; beyond material ranking.

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Abstract

Objective: To investigate the fundamental wear mechanisms of six resin-based composite (RBC) formulations during short-term *in-vitro* wear testing.

Materials: RBC materials were condensed into rectangular bar-shaped specimens and light irradiated using the ISO 4049 specimen manufacture and irradiation protocol. Wear testing (n=10 specimens for each RBC) was performed on a modified pin-on-plate wear test apparatus and wear facets were analysed for wear volume loss using a white light profilometer. The wear tested RBC specimens and their corresponding antagonists were analysed using scanning electron microscopy (SEM) and energy-dispersive X-ray spectroscopy (EDS), respectively to determine the wear mechanism.

Results: Data generated using the profilometer showed variations in the mean total wear volume (mm^3) between the RBCs tested ($p<0.05$). Abrasive wear was evident in all RBCs investigated with varying degrees of damage. Material transfer/deposition of the filler particles on the corresponding antagonists was evident in two RBC materials (Filtek Supreme and Kalore) indicative of a further adhesive wear mechanism.

Conclusion: It is proposed that the approach employed to use a combination of measurement and analytical techniques to quantify the wear facet volume (profilometry), wear trough (SEM) and material transfer (EDS) provides more useful information on the wear mechanism and the tribology of the system rather than relying on a simple wear ranking for the RBC materials as is routinely the case in dental research studies.

Introduction

The assessment of the wear performance of dental resin-based composite (RBC) restoratives has been determined frequently in the dental literature since the first *in-vitro* studies were published [1-2]. Today using the identifiable Medical Subject Headings (MeSHs) of 'dentistry AND wear', almost 2000 manuscripts have been published in the dental literature in the last in the last 10 years. To complicate wear performance data interpretation, a variety of *in-vitro* wear testing devices have been advocated to replicate the *in-vivo* masticatory process [3]. However, no single *in-vitro* wear simulator available can simulate the masticatory cycle in the oral environment [4]. At best wear simulators can provide an indication of the relative ranking of potential novel dental RBC restorative formulations prior to market launch when compared with commercially successful formulations [5-6]. Variations in RBC materials arise from different manufacturing processing routes and RBCs often include different monomeric resin matrices, functioning silane coupling agents and filler technologies (filler volume fractions, particle size distribution and filler density) [5-6]. However, the most robust laboratory RBC wear studies in the literature are conducted on a range of commercial dental products, routinely from different manufacturers in the form of round-robin tests [7-9].

Until recently, confusion existed on whether wear depth, area or volume should be reported [10] although the volume of material removed due to the interaction of opposing surfaces was shown to be the parameter of choice for reporting the *in-vitro* wear of RBCs [11] based on Archard's equation [12]. Too frequently in dentistry, wear depth or wear area are reported but wear in the mouth is dependent upon occlusal factors which change continuously with time and the progression of wear [10]. In addition, authors that claim to assess the wear volume

often fail to examine the wear facet sufficiently [13] or ensure the accuracy and precision of the wear measurements reported [5-6,11,14]. From a tribology perspective, there are four fundamental wear mechanisms that can exist, namely abrasion, adhesion, fatigue or corrosion [15-16] and wear facets are infrequently assessed following testing to elucidate the wear mechanisms operative during testing.

The aim of the current study was to investigate the short-term *in-vitro* wear resistance and wear mechanism operative during testing six RBC formulations. The null hypotheses stated were that there would be no differences in the (1) *in-vitro* mean total wear volume data and (2) wear mechanisms operative, for the commercial RBC formulations investigated.

Materials and methods

Materials

Six commercially available RBC materials with innovative claims in terms of monomer chemistry, filler content, filler type and/or filler size and produced by a range of dental manufacturers, for both anterior and posterior clinical use were selected (Table 1).

Specimen manufacture

The RBC materials was condensed into rectangular bar-shaped specimens (25.0 ± 0.1 mm length, 10.0 ± 0.1 mm width and 3.0 ± 0.1 mm thickness) using a custom made Perspex holder. A constant excess of uncured resin was placed into the mould, covered with a cellulose acetate strip and a glass microscope slide and a weight of 1 kg was applied for 20 s to ensure consistent and reproducible packing of the specimens. The weight and microscope slide were removed and the specimen was light irradiated using a light emitting diode (LED) light curing unit (LCU) (Demi Plus, Kerr, Orange Co., CA, USA) at ambient room temperature (23 ± 1ºC) with a spectral range of 450 - 470 nm and an irradiance of 1200 mW/cm^2. The irradiance was checked prior to use by employing a checkMARK (Bluelight Analytics Inc., Halifax, Canada). The entire length of each specimen was light irradiated using the ISO 4049 specimen manufacture protocol by placing the tip of the light guide in direct contact with the cellulose acetate strip in the centre of the specimen [17]. Both the top and the lower surface of the specimens were light irradiated to produce six groups of 10 specimens by overlapping the exit window by half the LCU tip diameter along the specimen [17] so that areas received twice the irradiation of adjacent areas using the 8 mm LCU tip diameter.

Following light irradiation, the cellulose acetate strip was discarded, the mould dismantled and the specimen removed and checked for surface imperfections. The specimens were wet ground by hand lapping using P400, P600, P800, P1000 and P1200 grit silicon carbide (SiC) abrasive papers (Struers, Copenhagen, Denmark) under copious water irrigation to remove the oxygen inhibited, resin rich layer and produce a planar surface with a consistent surface topography. The specimens were stored in a light-proof container and placed in a water-bath maintained at 37 ± 1ºC for seven days prior to testing and analysis.

Wear testing and analyses

To facilitate wear testing of contemporary RBC's, a newly modified pin-on-plate wear test apparatus developed originally by Harrison and colleagues [18-19] was used. The schematic in Figure 1 illustrates a cross section cut through one of the ten wear stations where a custom made antagonist holder was devised that could be attached underneath the vertical rod, to hold the abrader with the aid of locking screws. The modification to the original pin-on-plate wear test apparatus allowed for the choice of antagonist to be selected by the operator while the load used was maintained in line with masticatory forces. Differences between the original and the modified pin-on-plate wear testing apparatus are detailed in Table 2. The steatite sphere (8 mm diameter) wear antagonist [20] was fixed to the vertically moving pins and a loading force of 4.5 N was used [18] in a neutral buffer solution to approximate the *in-vivo* oral environment [21]. The RBC specimens were confined within a Perspex template attached to a horizontal plate moving at a frequency of 2.14 Hz.

Employing the modified pin-on-plate wear test apparatus, a pilot study was carried out to determine the minimum number of cycles to produce a linear wear rate with measurements following 2000, 3000, 4000 and 5000 wear cycles. Based on the results of the preliminary

study which showed a linear wear rate following 2000 cycles (r^2=0.99) (Figure 2), it was decided that all RBC materials should be tested for 4000 cycles - the equivalent of three months simulation in the oral cavity [18].

Profilometry

Wear tested samples displayed characteristic shallow wear tracks and were scanned using a TalySurf CLI 2000 profilometer (Taylor-Hobson Precision, Leicester, England) equipped with a with a 300 µm range chromatic length aberration (CLA) gauge scanning at 2 mm/s. Longitudinal traces were taken at 4 µm intervals (x-direction) across the wear facet with a measurement recorded at every 40 µm interval (y-direction) thereby generating a three dimensional (3D) profile (Figure 3) using the TalyMap Gold analysis software Version 4.2 (Taylor-Hobson Precision, Leicester, England). The unworn areas around the wear track were used as the datum from which it was possible to calculate both the mean maximum wear depth and the mean volume loss (mm^3) of the RBC materials investigated [5-6,11].

In line with the profilometic analyses, ten traces were performed across a standard step height of 1.0 mm to determine the accuracy and precision of the wear depth measurements for the scanning conditions (300 µm range CLA, scanning at 2 mm/s with longitudinal traces at 4 µm intervals (x-direction) and horizontal traces recorded at 40 µm intervals (y-direction) for a resolution of 0.1 µm (z-direction). The accuracy was calculated as the mean error from the true value, whilst the precision was quantified as the standard deviation of the errors measured [5,10-11].

Scanning electron microscopy (SEM) and Energy-dispersive X-ray spectroscopy (EDS)

The wear tested RBC specimens and their corresponding antagonists were mounted on aluminium stubs and sputter coated with approximately 5 nm of gold using an argon sputter coating unit (Agar Scientific, Stanstead, UK). The samples were analysed using a Hitachi-S-3400N, variable pressure scanning electron microscope (Hitachi High-Tech Technologies, Tokyo, Japan) under low vacuum at a 5 mm distance to elucidate the wear mechanism operative. Additionally, Energy-Dispersive X-ray Spectroscopy (EDS) (Bruker Inc., Berlin, Germany) analyses were conducted on the steatite antagonists to record the elemental spectral maps to further elucidate the wear mechanism operative.

Statistical analyses

All data sets were checked for normality using a Kolmogorov–Smirnov test and Shapiro-Wilk test. Data were analysed by Kruskal-Wallis Test with Post Hoc Bonferroni with a significance level of $p<0.05$.

Results

Wear Analyses

The accuracy and precision for the 1.0 mm step size was 1.51 and 0.54 µm, respectively. Data generated using the profilometer showed variations in the mean total wear volume (mm^3) for the six RBCs tested (Table 3) with the mean total wear volume identified to be significantly higher for Filtek Supreme compared with the other RBC materials tested ($p<0.001$). Further statistical analyses revealed Kalore also showed higher mean total wear volume values compared with Venus Diamond and Clearfil Majesty (all $p<0.05$). However, no significant differences in mean total wear volume were observed between Filtek Silorane, Venus Diamond, Tetric Ceram HB and Clearfil Majesty ($p>0.05$).

Qualitative SEM analyses of the wear facets highlighted abrasive wear in all RBC restoratives under investigation with varying degrees of damage including pitting, cracking and material loss (Figure 4). SEM analyses identified similar wear tracks (prominent vertical grooves), the opposing steatite antagonist had round and epileptically shaped wear facets of variable sizes (Figure 5).

EDS analyses of the steatite antagonists confirmed than in addition to the expected magnesium and silicon present from the steatite ($Mg_3Si_4O_{10}(OH)_2$), zirconia was present following the wear testing of Filtek Supreme and aluminium was present in the analyses of the anatagonists for Kalore (Figure 6). Both zirconia and aluminium are the major filler components in the Filtek Supreme and Kalore RBCs, respectively which suggests material transfer and deposition of the filler particles on the corresponding antagonists indicative of an adhesive wear mechanism being operative.

Discussion

In-vitro wear testing of RBCs routinely results in abrasive wear, manifest when the steatite antagonist contacts the RBC surface, causing a ploughing action [15-16], resulting in plastic deformation with the removal of RBC material to cause a wear track. The SEM analyses of the wear facets highlighted abrasive wear in all RBC groups with varying degrees of damage - pitting, cracking and material loss (Figure 4) depending upon the filler technology employed (filler volume fractions, particle size distribution and filler density). Previous studies have reported that increasing the filler volume fraction increases the *in-vitro* wear resistance [22-25]. However, it should be noted that these studies [22-25] were performed on older RBC materials with lower filler volume fractions where a marked increase in filler volume fraction would be expected to make a substantial difference to the *in-vitro* wear resistance. Finlay et al. [6] identified the *in-vitro* wear resistance of experimental RBCs provided by a dental manufacturer, where the RBC compositions were tailored by adjusting the filler volume fraction, diameter or density, and/or the resin monomeric blend [26-27], to be dominated by the filler constituent, although wear was described as a 'complex process' and not all resin formulations were reported to behave similarly [6]. This statement holds true for the two RBC materials (Filtek Supreme and Kalore) which exhibited exacerbated mean total wear volume data compared with the four other RBC formulations and this was due in a significant part to the presence of a second adhesive wear mechanism (Figure 5). Adhesive wear results in the transferral of material from the RBC to the steatite antagonist by cold welding through friction [15-16, 28]. It is suggested that the *combination* of abrasive and adhesive wear mechanisms during the testing of Filtek Supreme and Kalore were responsible for the exacerbated mean total wear volume values reported compared with the four RBC formulations where only the abrasive wear mechanism was evident. Statistically, no

significant differences in the mean total wear volume were evident in these four RBC materials following three months simulation in the oral cavity. This result was not surprising despite the numerous material factors (monomeric blend, filler volume fraction, mean filler diameter and filler density) being different in the RBCs tested. Tetric Ceram HB contained pre-polymersied filler particles present as large agglomerates within the matrix and SEM images of the wear track showed evidence of loss of large individual filler particles corresponding to the agglomerates (Figure 4C). However the volumetric wear loss was not significant compared with the other conventionally filled RBCs. Whilst short-term wear of RBCs (equivalent of 6 month *in-vivo*) was shown previously [6] to be not as well able to discern between RBC formulations compared to extended wear testing (equivalent of 18 and 36 months *in-vivo*) it did provide significant insights into the *in-vitro* wear behaviour of the RBC formulations investigated [6] and the same was evident in this study. Therefore it is proposed that further insights into the *in-vitro* wear behaviour of the RBC formulations that were found to exhibit abrasive wear only could be obtained following an extended wear test. The profilometric analyses were performed across an area of 8 mm length and a 3 mm width with data points recorded every 40 µm interval in the y-direction and every 4 µm in the x-direction, resulting in 150,951 data points for each wear facet which increases the confidence in the mean total wear volume data [5,11] compared with analogue measurements routinely used in dentistry [7-9,13,29-33]. Additionally, the accuracy and precision volumetric loss measurement data was confirmed by identifying for the accuracy and precision of data recorded for a 1.0 mm step size which was 1.51 and 0.54 µm, respectively.

The overarching aims of this study were to investigate the short-term *in-vitro* wear resistance and wear mechanisms operative during wear testing, as such the choice of the six RBC's was somewhat unimportant, a range of contemporary materials with claimed novelty in

either/both filler/matrix were chosen to be representative of those currently available. Both null hypotheses that there would be no differences in the (1) *in-vitro* mean total wear volume data and (2) wear mechanisms operative, for the six RBC formulations investigated were rejected.

The oral wear simulator used was a variant of the device developed by Harrison and Lewis [18] to simulate the intermittent sliding action of teeth which remains a major step forward from the conventional pin-on-disc devices routinely used to study wear in engineering materials [3]. Of the *in-vitro* wear testing devices advocated to replicate the masticatory process [3] none can truly simulate *in-vivo* wear data. This is in part due to 'semi-quantitative methods for assessing the wear *in-vivo* on pooled data from clinical trials' [9].

Ferracane (2013) [34] has identified that wear resistance should be evaluated as a screening tool when selecting an RBC to replace occlusal surfaces. What is significant in the approach employed in the current study is the use of a combination of measurement and analytical techniques to quantify the wear facet (profilometry), wear trough (SEM) and material transfer (EDS) to provide useful information on the mean total wear volume, wear mechanism operative and therefore the tribology of the system rather than relying on a simple wear ranking of RBC materials. If the current approach to focus on the tribology of the RBC system was adopted by other researchers then there is an increased likelihood that substandard RBC materials that wear by a combination of abrasive and adhesive wear processes could be easily identified and their clinical use limited for patients with an increased wear risk.

Conclusion

The use a combination of measurement and analytical techniques to quantify the wear facet (profilometry), wear trough (SEM) and material transfer to the antagonist (EDS) provides more useful information on the wear mechanism and the tribology of the system rather than relying on a simple wear ranking for the RBC materials as is routinely the case in dentistry.

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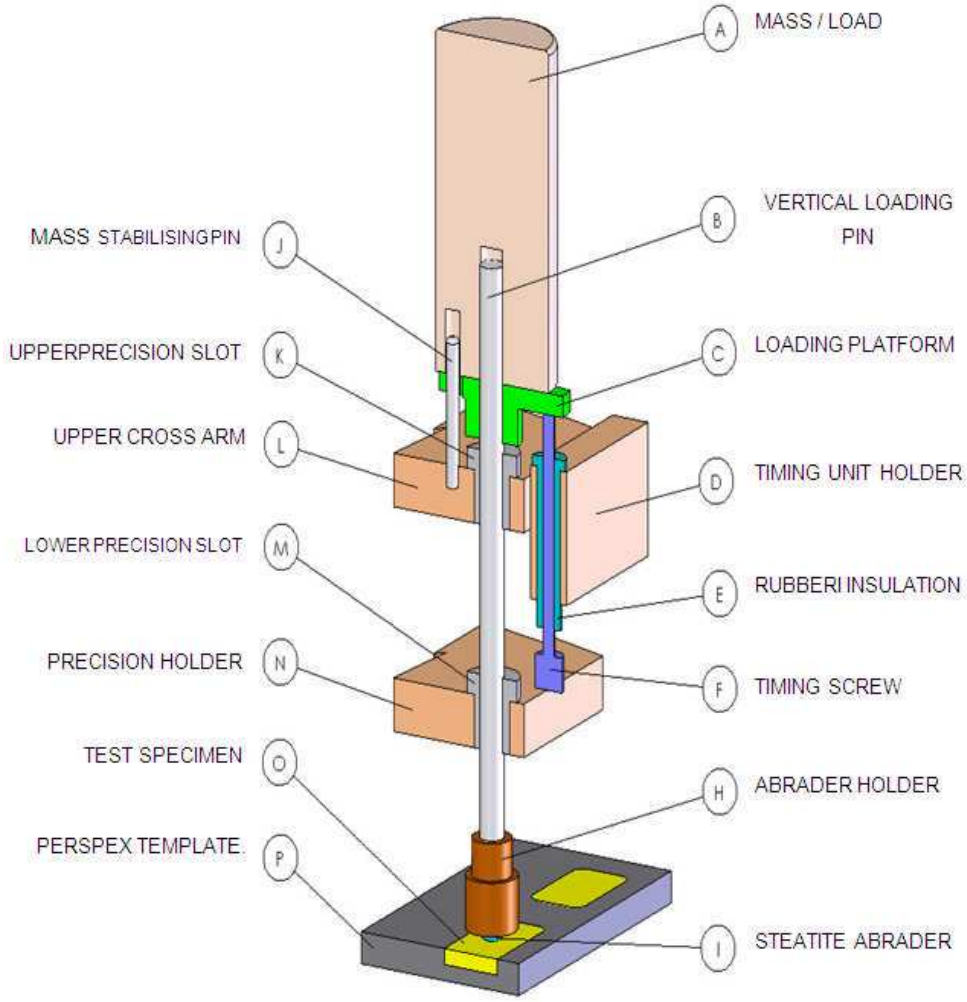

Figure 1: Schematic illustrating a cross section cut through one of the ten wear stations where a custom made antagonist holder was devised that could be attached underneath the vertical rod.

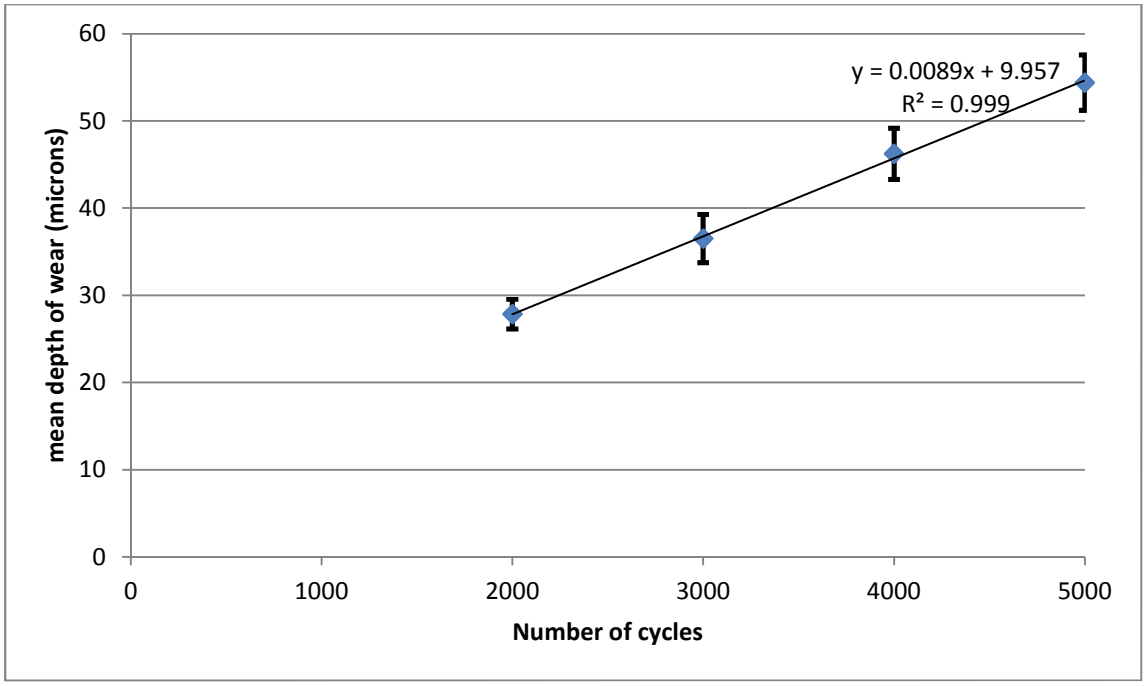

Figure 2: Data from the pilot study to determine the number of wear cycles necessary to produce a linear wear track with measurements taken at 2000, 3000, 4000 and 5000 wear cycles.

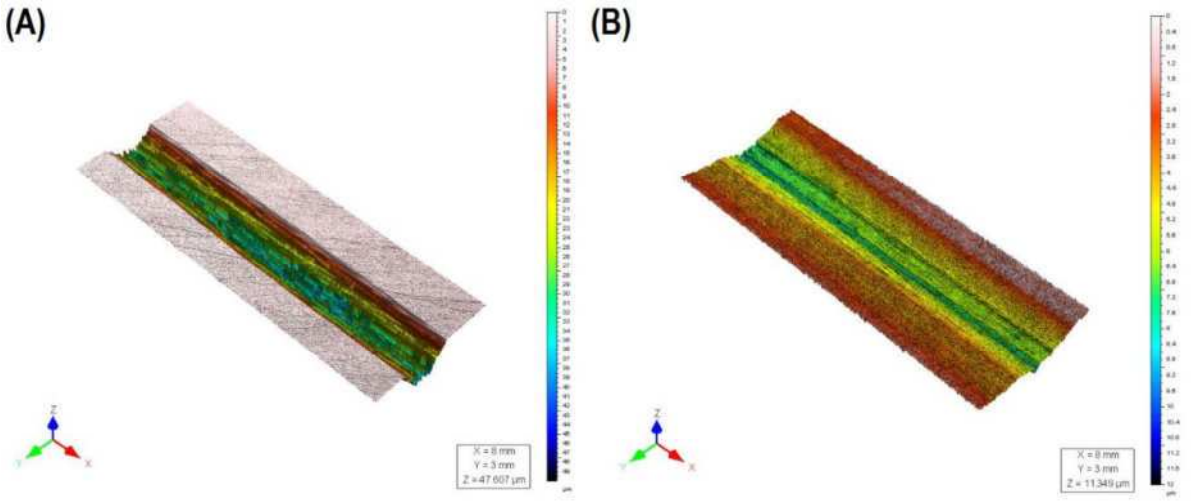

Figure 3: Profilometric scans showing examples of a deep wear track in (A) for Filtek Supreme XTE sample and (B) shallow wear track for a Clearfil Majesty Posterior sample.

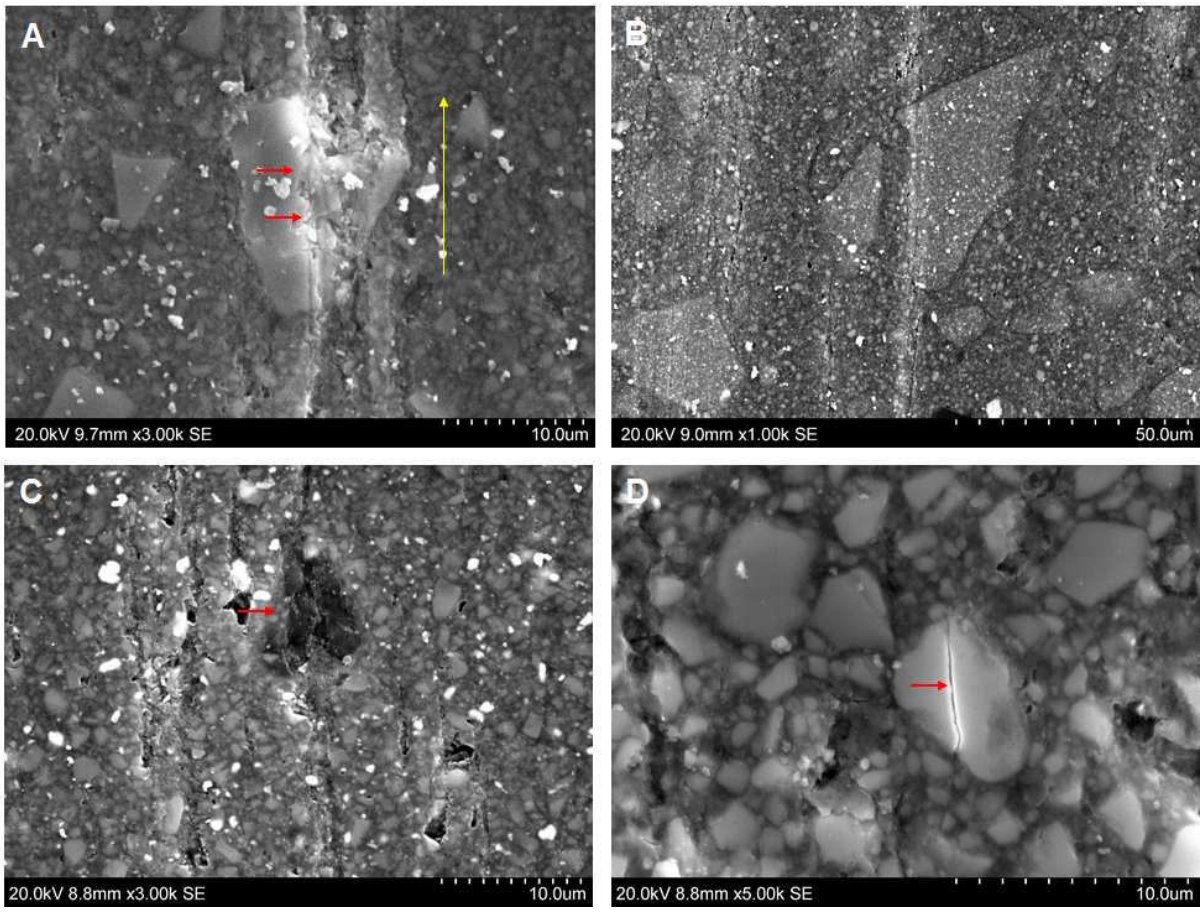

Figure 4: Scanning electron micrographs highlighting abrasive wear for (A) Venus Diamond sample, showing abrasive wear on a filler particle (red arrows), yellow arrow illustrate the direction of wear, (B) Kalore sample with the wear track cracking a filler particle, (C) Tetric Ceram HB sample with a removed large pre-polymerised filler particle (red arrow), (D) cracked filler-particle (red arrow) in the direction of wear in a Clearfil Majesty Posterior sample.

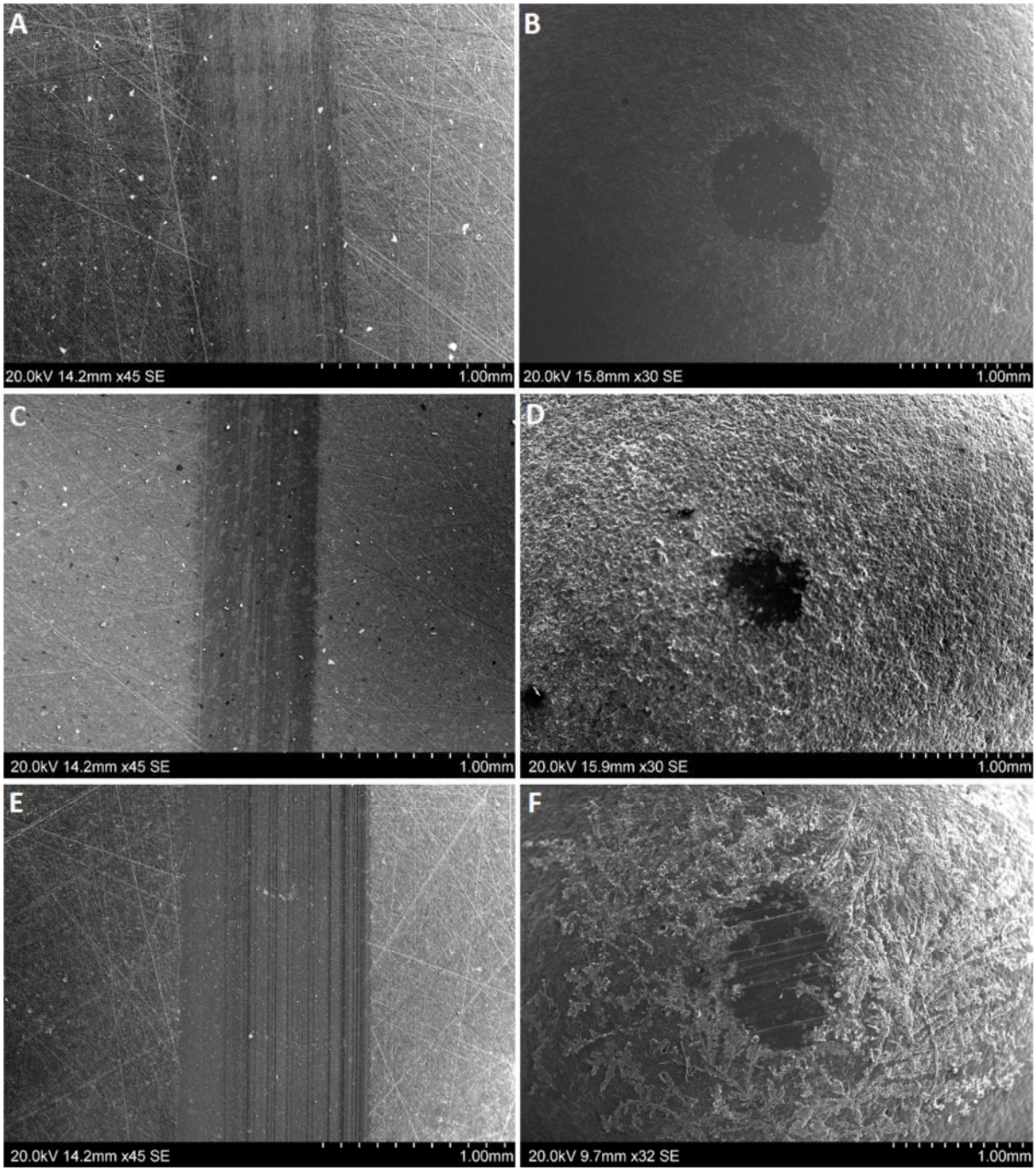

Figure 5: Scanning electron micrographs highlighting the RBC samples and the corresponding steatite antagonist for Filtek Silorane (A and B), Kalore (C and D) and Filtek Supreme XTE (E and F).

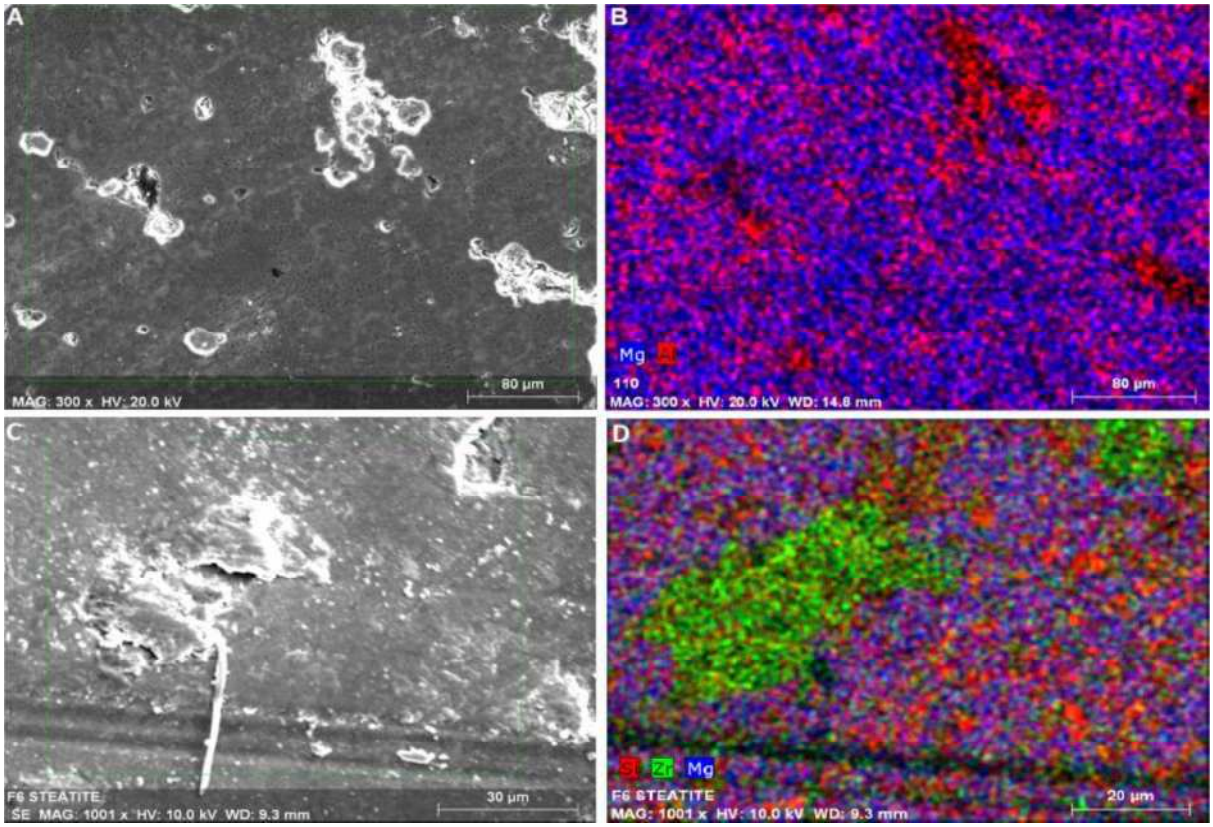

Figure 6: Scanning electron micrographs highlighting a steatite antagonist opposing (A) a Kalore sample and (C) a Filtek Supreme sample. Energy-dispersive X-ray spectroscopy analysis showing aluminium transfer on the steatite antagonist, opposing (B) a Kalore sample and zirconia particles deposited on the steatite antagonist of (D) a Filtek Supreme sample.

Table 1: Manufacturers details for the six commercially available RBC materials selected.

RBC	Description	Manufacturer	Resin	Filler type/size	Content	Special characteristics
Filtek Silorane	Universal Microhybrid	3M ESPE, USA	*Silo*xanes and Oxi*ranes*	Quartz, YF 0.1-0.2µm	76wt% 55vol%	Ring-opening monomers.
Filtek Supreme	Universal nanofilled	3M ESPE, USA	BisGMA,$BisEMA_6$ UDMA,TEDMA PEGDMA	ZrO_2, SiO_2 0.6–1.4 µm	72 wt% 55 vol%	"True" nanotechnology unique clusters of nano-sized particles.
Kalore	Universal nanohybrid	GC America, USA	UDMA DX-511 co-monomers, Dimethacrylate	F-Al-Si, SiO_2 0.4-0.7µm	82 wt%	Does not contain BisGMA, DuPont's new monomer* technology
Venus Diamond	Universal nanohybrid	Heraeus Kulzer Hanau, Germany	TCD-DI-HEA, UDMA	Ba-Al-F, SiO_2 0.5nm-20µm	65 wt% 41vol %	New cross linker technology. The TCD-urethane cross linker.
Tetric Ceram HB	Universal nanohybrid	Ivoclar-Vivadent Liechtenstein	BisGMA, UDMA, BisEMA	Ba–F–Al–B–Si mixed oxides, SiO2, YbF_3, PPF 0.4-1µm	76wt% 55vol%	Containing BisEMA Monomer
Clearfil Majesty Posterior	Universal nanofilled	Kuraray, USA	BisGMA, TEGDMA	Alumina and glass-ceramic 20nm-1.5µm	92wt% 82vol%	Nano Dispersion Technology High filler content.

BIGMA: Bisphenol A diglycidal ether dimethacrylate, TEGDMA: Tri ethylene glycol dimethacrylate, BISEMA: Bisphenol A polyethylene glycol diether dimethacrylate, $BISEMA_6$: Hexa ethoxylated Bisphenol A polyethylene glycol diether dimethacrylate, PEGDMA: Poly ethylene glycol dimethacrylate, UDMA: Urethane di methacrylate, TCD-DI-HEA: 2-propenoic acid, (octahydro-4,7 methano-1H-indene-5-diyl) bis(methyleneiminocarbonyloxy-2,1-ethanediyl) ester, PPF: Pre-polymersied fillers.

Table 2: A detailed comparison of the differences between the original pin-on-plate wear test apparatus developed by Harrison and colleagues and the modified pin-on-plate wear testing apparatus used in the current study.

Variable	Original pin-on-plate wear test apparatus	Modified pin-on-plate wear testing apparatus
Abrader	Silicon carbide paper held individually in the table.	Steatite antagonist, 8 mm diameter but can be modified to fit any diameter.
Test sample	cylindrical specimens (4.5 mm diameter) cemented onto pin ends.	Rectangular-bar shaped specimens (25.0 x 15.0 x 3.0 mm) held in the table with locking screws.
Number of test specimens	10	10
Pin plate contact frequency	70/min	100/min
Pin plate contact time	0.2 s but can be adjusted for each sample	0.2 s but can be adjusted for each sample
Pin plate vertical lift	4 mm	4 mm
Pin plate contact distance	1 mm	1 mm
Stroke frequency	2.10 Hz	2.14 Hz
Environment	Liquid or slurry abrasive	Liquid or slurry abrasive
Measurement of wear	Vertical height loss of specimen using a specially designed bench micrometer.	Maximum depth (mm) and/or volume loss (mm^3) using a non-contact profilometer.
Masses used	50 – 1000 g	50 – 1000 g

Table 3: Mean volume wear loss and associated standard deviations (mm^3) for the six RBCs investigated.

RBC	Volume Loss (mm^3)	
	Mean	**Std. Deviation**
Filtek Silorane (SI)	0.030	0.009
Filtek Supreme (FS)	0.354	0.091
Kalore (GC)	0.049	0.010
Venus Diamond (VD)	0.012	0.002
Tetric Ceram (TC)	0.022	0.003
Clearfil Majesty CM)	0.021	0.001