Australian Dental Journal
The official journal of the Australian Dental Association

Australian Dental Journal 2023; 0: 1–15

doi: 10.1111/adj.13004

Comparative analysis of self-cure and dual cure-dental composites on their physico-mechanical behaviour

J Thadathil Varghese,[*] R Raju,[†] P Farrar,[‡] L Prentice,[‡] BG Prusty[*,†,§]

*School of Mechanical and Manufacturing Engineering, University of New South Wales, Sydney, New South Wales, Australia.
†ARC Centre for Automated Manufacture of Advanced Composites, School of Mechanical and Manufacturing Engineering, University of New South Wales, Sydney, New South Wales, Australia.
‡SDI Limited, Bayswater, Victoria, Australia.
§Australian Composites Manufacturing CRC Ltd. (ACM CRC), University of New South Wales, Sydney, New South Wales, Australia.

ABSTRACT

Background: Clinical practitioners may have become familiar with the rapid transformation of dental composites. However, they may not scientifically understand the factors influencing the mechanical and physical properties. Scientific knowledge of filler-resin interaction can significantly improve clinical understanding of resin composites. Several independent studies have examined the mechanical and physico-mechanical properties of dental resin composites; however, no comprehensive study has examined the influence of fillers and resin materials on the physico-mechanical properties of both self-cure and dual-cure composites.
Methods: This study performed investigations on the physico-mechanical behaviour of four commercially available dual-cure dental composites (Bioactive, Fill Up!, Surefil One, Cention N) and two commercially available self-cure dental composites (Stela Capsule and Stela Automix). Test specimens for flexural and compressive strength, microhardness, fracture toughness, and hydrolytic behaviour were prepared and tested as per respective standards. The data sets were statistically analysed using one-way ANOVA and Tukey's post-hoc comparison.
Results: There was a substantial variation in flexural strength and modulus values in this study, ranging from 32.0 to 113.4 MPa and 2.36 to 12.07 GPa, respectively. Similarly, there were significant differences in compressive strength between the materials in this study, ranging from 119.3 to 223.5 MPa. The highest fracture toughness value was found to be 1.41 $\mathrm{MPa.m^{0.5}}$, while the lowest value was 0.43 $\mathrm{MPa.m^{0.5}}$. Variations in surface microhardness were significant (24.11–68.0 $\mathrm{N/mm^2}$), which correlated with the filler content. Water sorption and solubility demonstrated high variations among materials, with Surefil One exceeding ISO 4049 thresholds significantly.
Conclusions: A linear correlation can be established between surface microhardness (HV) and flexural and compressive moduli, as well as filler content (wt.%). However, both flexural and compressive strengths are impacted by the resin's constituent monomers and the resin-filler matrix's cross-linking capability. Additionally, factors such as filler size, shape, and the cross-linking ability of the resin-filler matrix play a crucial role in fracture toughness and the propagation of cracks within the restoration. Also, resin monomers and filler particle size affect the hydrolytic degradation characteristics of composites, which can also affect their mechanical properties. © 2023 Australian Dental Association.

Keywords: Dental composite, physico-mechanical properties, self-cure, dual-cure, hydrolytic degradation, commercial composite, filler-resin interaction.

Abbreviations and acronyms: UDMA = Urethane Dimethacrylate; GDMA = Glycerol Dimethacrylate; YbF_3 = Ytterbium Trifluoride; TEGDMA = Triethylene Glycol Dimethacrylate; Bis-GMA = Bisphenol-A Glycol Dimethacrylate; TMPTMA = Trimethyl Propane Trimethacrylate; CQ = Camphorquinone; Ste_Mix = SDI Stela Automix; Ste_Cap = SDI Stela Capsule; Act_Bio = Activa Bioactive; Fill_Up = Fill Up!; Sur_One = Surefil One; Cen_N = Cention N; DRC = Dental resin composites; MI = minimally invasive; UV = ultraviolet; VH = Vickers hardness; W_{sp} = water sorption; W_{sl} = solubility.

(Accepted for publication 30 November 2023.)

CLINICAL RELEVANCE

This study investigates the physico-mechanical properties of six newly available commercial dental resin composites that have not previously been published in the literature. Using this study, dental practitioners and researchers can gain a deeper understanding of the performance of restorative materials and how filler content, resin type, and mechanical properties are related. Clinicians will, therefore, be able to make a more informed selection of materials appropriate to the specificity of

cavity sizes and locations. Additionally, the study explores adhesion, crack propagation, and water sorption, providing practical insight into enhancing material durability and longevity in clinical applications.

INTRODUCTION

Approximately one-third of the population, regardless of age group, is affected by tooth decay. There are various methods of restoring these decays, including resin-based dental composites (44%), amalgams (40.9%), glass ionomers/resin-modified glass ionomers/dental composites (13.4%), and other methods (e.g., indirect and temporary restorations) (1.7%).[1] According to the statistics provided above, the use of resin-based dental composites in place of amalgam is on the rise in the dental field. It is important to note that the current restorative composites have superior mechanical and physical properties. However, they only last approximately ten years, after which professional intervention may be necessary.[2] It has been reported that recurrent caries and restorative bulk fractures are the most common causes of clinical failure.[2] There was a 56% replacement rate for dental restorations placed before 2000 and a 58% replacement rate for dental restorations placed after 2000.[1] Approximately 5% of these restorations were anticipated to experience bulk fracture within 10 years, while 12% will exhibit significant wear.[2] A primary factor affecting the longevity of dental resin composites is the composition of the material.[1,2]

The properties of dental restorative materials present several challenges, and obtaining an ideal dental restorative material remains an ongoing research topic. The following properties are considered to be ideal properties of a restorative material: (a) mechanical properties, (b) application, (c) biocompatibility, and (d) aesthetics.[3] There have been advances in filler particles (amount, shape, or surface treatment), monomer chemistry, and altered polymerization dynamics to overcome the limitations of conventional resin-based materials and their associated restorative techniques.[4] A variety of curing modes to improve the polymerization dynamics for clinical dental resin composites (DRCs) have been developed by research, including self-cure, light-cure, and dual-cure DRCs.[5] While different techniques have been used to improve the mechanical and physical properties of DRCs, optimizing the interface interaction between fillers (inorganic) and resin matrix (organic) has proven to be one of the most successful strategies.[6] Another way to improve filler/resin interfacial interaction is by modifying the fillers, which further improves the comprehensive properties of DRCs.[6] However, with the increase in molecular weight of filler (which can enhance the mechanical properties[7]), light-cured composites suffer from high polymerization shrinkage (in the range of 2–6%).[8] This creates significant shrinkage stress, which may create microcracks, leading to restoration failure.[9]

Moreover, light-cured DRCs are often placed in increments of 2 mm to ensure complete light-induced polymerization. This is because the depth of curing lights has a limitation due to the light attenuation property.[10,11] Over time, bulk-filled composites have been developed to improve the curing depth, thereby permitting DRCs to be placed in larger increments of up to 4 mm.[12] However, these systems still rely on the depth of light penetration to activate the polymerization process. Moreover, incremental placement of DRCs could potentially introduce unwanted voids that would decrease the strength of the restoration.[13]

Considering this problem, chemically cured (self-cured) composite materials are being used as direct restorative materials due to their low shrinkage stress (as a result of low shrinkage, longer pre-gel phase, and slower polymerization) and infinite depth of cure.[21,22] In general, self-cured DRCs consist of resin with 70–80 wt.% concentrations of irregularly shaped and macro-sized (100 μm in diameter) quartz, borosilicate, ceramic, or glass particles, which initiates the curing process on mixing.[23] However, when hand-mixed, the monomers in self-cured DRCs could lead to mixing errors in paste concentrations and the inclusion of air bubbles in the polymer, resulting in compromised material properties.[23]

The dual-cured DRCs were designed to have quick and on-demand curing characteristics seen with light-cure DRCs and characteristics to overcome the lack of light accessibility in deep cavities.[14] Dual-cure DRCs include a chemical reaction to activate self-cure reaction from mixing two components, along with independent light curing with the help of photoinitiators.[15] Therefore, dual-cure DRCs have the potential to attain unlimited curing depths through the self-cure mode.[16,17] However, previous studies have shown that when dual-cure DRCs are underexposed to light, they may not obtain maximum mechanical properties, as the monomers do not achieve their maximum degree of conversion.[18,19]

Therefore, a comprehensive understanding of existing dental materials is necessary for developing and implementing new restorative materials[3] and for clinicians to select the best restorative materials based on the type of cavity. Even though a wide variety of DRCs are developed and employed as restorative dental materials, there is limited information regarding their comprehensive behaviour examining how material properties and self- and dual-curing modes affect material performance.

Therefore, the primary objectives of this study are to examine the relationship between filler content and flexural and compressive elastic moduli of dental restorative materials, as well as how resin type influences the strength of these materials. The study also aims to evaluate how fillers affect crack propagation and the critical role of adhesion between fillers and matrix in improving fracture toughness by analysing the effect of shape, size, and distribution on fracture toughness. The research also examines how filler content, in conjunction with the resin matrix, influences the surface hardness of dental restorative materials. Finally, the study intends to develop a predictive model based on filler content (wt.%) data to estimate flexural, compression, and surface microhardness in dental restorative composites. In addition, this study also examines the interaction of resin type and filler content with respect to water sorption and solubility in these materials.

MATERIALS AND METHODS

Materials

Six commercially available DRCs were prepared and evaluated *in vitro*. The composition and preparation techniques for each composite are detailed in Table 1. An LED curing light (Radii Plus, SDI Ltd, Australia) with an output of 1500 W/cm^2 was used as an external energy source for curing Type 1-Class 3 materials ACTIVA Bioactive (Pulpdent, USA), Fill Up! (Coltene, Switzerland), Surefil One (Dentsply Sirona, Germany), and Cention N (Ivoclar Vivadent, Liechtenstein). Two Type 1-Class 1[20] self-cure dental composites, namely Stela Automix (SDI Ltd, Australia) and Stela Capsule (SDI Ltd, Australia), were also used in this study. The flexural strength/modulus, fracture toughness, compressive strength/modulus, water sorption, solubility, and microhardness of the above-mentioned dual-cured and self-cured DRCs were characterized, and the mechanical/physical behaviour parameters were studied. The application technique for the commercially available dual-cure DRCs and self-cure DRCs is mentioned in Table 1.

Mechanical properties

Flexural properties

Three-point bend tests were conducted as per ISO 4049:2019 to measure the flexural properties of DRCs. For each group, six bar-shaped specimens of dimensions 25 ± 0.1 mm × 2.0 ± 0.1 mm × 2.0 ± 0.1 mm were prepared using a stainless-steel mould. The dual-cure dental composite samples (as shown in Table 1) were cured using overlapping irradiation[20] with blue light (Radii Plus, SDI Limited, Australia) with wavelengths ranging from 440 to 480 nm and intensity of 1500 mW/cm^2 for 60 s on each side of the samples. Both self-cure and dual-cure composite samples were chemically cured for six minutes, following previous studies performed by the authors and from the data obtained on commercial composites.[21,23] The cured specimens were stored in distilled water at 37 ± 1°C using a Thermo Scientific Heratherm compact microbiological incubator for 24 h and then polished using a wet 1200-grade silicon carbide paper before testing.[20,24] After a minimum of 24 h from the commencement of

Table 1. Description of the dental composites, manufacturers, type and class, composition, filler loading in wt.% and type of curing as obtained from the manufacturers[21–23]

Material	Manufacturer	Class	Composition	Filler loading	Curing
Ste_Mix	SDI	Type 1-Class 1	A blend of UDMA, GDMA, Fumed Silica, Barium Aluminoborosilicate Glass, Fluoro Aluminosilicate Glass, YbF_3, Calcium Aluminate, Initiators, Stabilizers, Pigments	61.0 wt.% 36.0 vol.%	Self-Curing
Ste_Cap	SDI	Type 1-Class 1	A blend of UDMA, GDMA, Fumed Silica, YbF_3, Fluoro Aluminosilicate Glass, Calcium Aluminate, Initiators, Stabilizers, Pigments	77.0 wt.% 55.0 vol.%	Self-Curing
Act_Bio	Pulpdent	Type 1-Class 3	A blend of diurethane dimethacrylate and other methacrylates, modified polyacrylic acid, silica, amorphous, sodium fluoride	56.0 wt.% NA vol.%	Dual-Curing
Fill_Up	Coltene	Type 1-Class 3	TMPTMA, UDMA, bis-GMA, TEGDMA, benzoyl peroxide, Zinc oxide coated	65.0 wt.% 49.0 vol.%	Dual-Curing
Sur_One	Dentsply Sirona	Type 1-Class 3	Aluminium-phosphor-strontium-sodium-fluoro-silicate glass, highly dispersed silicon dioxide, YbF_3, Polycarboxylic acid, bifunctional acrylate, acrylic acid, iron oxide pigments, water, titanium dioxide pigments, CQ, stabilizer, self-cure initiator	77.0 wt.% NA vol.%	Dual-Curing
Cen_N	Ivoclar Vivadent	Type 1-Class 3	Liquid: dimethacrylates (UDMA, DCP, and an aromatic aliphatic-UDMA and PEG-400 DMA), initiators, stabilizers, and additives. Powder: Calcium fluoro-silicate glass, Barium glass, Calcium-barium aluminium fluoro-silicate glass, ISO fillers, YbF_3, initiators, and pigments	78.4 wt.% 57.6 vol.%	Dual-Curing

mixing or irradiation, the specimens were loaded into an Instron 3369 Universal Testing Machine and supported by two rods with a diameter of 2 mm and separated by 20.0 ± 0.1 mm. Displacement load was applied at a crosshead speed of 1 mm/minute until fracture. The flexural strength (σ_f) and flexural modulus (E_f) were evaluated as

$$\sigma_f = \frac{3FL}{2bd^2}\ MPa \tag{1}$$

$$E_f = \frac{L^3 y}{4bd^3}\ GPa \tag{2}$$

where F is the maximum load in N, L is the distance between supports in mm, b is the width of the specimen in mm, y is the gradient of the initial straight-line portion of the load-deflection curve, and d is the depth or thickness of the tested specimen in mm.[9,20]

Compressive properties

Compressive tests were performed to measure the compressive strength of DRCs. For each group, five cylindrical-shaped specimens of dimensions 6.0 ± 0.1 mm high and 4.0 ± 0.1 mm diameter were prepared using an aluminium mould.[25,26] Curing self-cure and dual-cure dental composites is performed as mentioned earlier in Section 2.2.1. The cured specimens were also stored in distilled water at 37 ± 1°C for 24 h and polished before testing. Displacement load was applied at a crosshead speed of 1.00 mm/minute until fracture. Compressive strengths (σ_c) were evaluated based on the following formula, and the compressive moduli (E_C) were evaluated based on the analysis of compressive stress-strain plots[26]:

$$\sigma_c = \frac{4F}{\pi D^2}\ MPa \tag{3}$$

where F is the maximum load in N, and D is the diameter of the tested specimen in mm.

Fracture toughness

Five samples per group with dimensions of 25.0 ± 0.1 mm × 5.0 ± 0.1 mm × 3.0 ± 0.1 mm and a pre-crack depth of 2.50 ± 0.25 mm were prepared using a stainless-steel mould and a razor blade.[27] Self-curing and dual-curing dental composites are cured as described earlier in 2.2.1. The cured specimens were stored in distilled water at 37 ± 1°C for 24 h and polished using a wet 1200-grade silicon carbide paper and then stored in distilled water at 37 ± 1°C using a Thermo Scientific Heratherm compact microbiological incubator for 24 h before testing. Displacement load was applied at a crosshead speed of 0.50 mm/minute until fracture. For each specimen, a Keyence VK-X200 3D Laser Scanning Microscope was used to measure the pre-crack depth on the fracture surface. The fractured surface morphology was observed using a Hitachi TM4000 Plus Tabletop Scanning Electron Microscope (SEM). The maximum stress intensity factor (K_{1C}) of DRCs was calculated per ISO 20795-1 and is given by the following formula.[25,27]

$$K_{IC} = \frac{P_{\max} S}{b h^{3/2}} f\left(\frac{a}{h}\right) \times \sqrt{10^{-3}}\ MPa.m^{1/2} \tag{4}$$

where $P_{\max}$ is the fracture load in N, S is the span length of the specimen in mm. h, b and a are the height, width, and crack length of the specimen, respectively, in mm. The $f(a/h)$ is a geometrical function given by

$$f(a/h) = 3(a/h)^{1/2}\left[\frac{1.99-(a/h)(1-(a/h))\left[2.15-3.93(a/h)+2.7(a/h)^2\right]}{2(1+2(a/h))(1-(a/h))^{3/2}}\right] \tag{5}$$

Microhardness

Hardness ensures that the placed restorations resist mastication forces and abrasion processes. 12 specimens per group with a dimension of 10.0 ± 0.1 mm × 5.0 ± 0.1 mm × 3.0 ± 0.1 mm were prepared using stainless steel mould. As mentioned in 2.2.1, self-curing dental composites are cured by chemical curing, while dual-curing dental composites are cured using UV light. The specimens were polished using 1200-grade silicon carbide paper. The Vickers hardness (VH) tests were performed on a Struers DuraScan-80 Hardness Tester per ISO 6507-1.[28] Ten indentations were placed on each specimen surface by a 136° pyramidal diamond indenter with a load of 500 g and a dwell time of 5.0 s. The minimum distance between indentations was kept $>3d$, where d is the average diagonal length of the indentation. The following expression gives the VH:

$$VH = 0.1891 \times \frac{F}{d^2}\ N/mm^2 \tag{6}$$

where d^2 is the indentation area, and F is the applied load in N.

Physical properties

Water sorption and solubility

Six disc-shaped specimens per group ($N = 6$) with dimensions 1.0 ± 0.1 mm thick and 15.0 ± 0.1 mm diameter were prepared using a stainless-steel mould for water sorption and solubility tests.[20] The volume V of each specimen was evaluated from their thickness and diameter. To cure dual-cure dental composites, eight overlapping sections of the sample were photopolymerized for 20 s on both sides,[20] using blue light (Radii Plus, SDI Limited, Australia). The self- and dual-cure composites were allowed to chemically cure for six minutes, as mentioned in 2.2.1. All cured specimens were dried in a desiccator with dry silica gel for 24 h at 37 ± 1°C and then transferred to the second desiccator with dry silica gels and maintained at 23 ± 1°C for 2 h. The mass (m_1) of each specimen was measured using an analytical balance with an accuracy of 0.1 mg (Ohaus PA214). This cycle was repeated every 24 h until no more than 0.001 g of weight loss within 24 h. The dried specimens were immersed in distilled water and maintained at 37 ± 1°C for seven days. After seven days of immersion, the specimens were removed from the distilled water, wiped, and waved in the air for 15 s, and the mass of each specimen was recorded as m_2. The drying cycle was repeated until they reached the constant mass, recorded as m_3. The water sorption and solubility of DRCs were evaluated following ISO 4049:2019.[20] The expressions for water sorption (W_{sp}) and solubility (W_{sl}) are given by

$$W_{sp} = \frac{m_2 - m_3}{V}\ \mu g/mm3 \quad (7)$$

$$W_{sl} = \frac{m_1 - m_3}{V}\ \mu g/mm3 \quad (8)$$

Surface morphology

The fractured surfaces after the fracture toughness tests were used for image analysis of the surface of the tested groups. Before testing, the specimens were mounted onto the aluminium stubs with a fast-setting adhesive. The specimens were then sputter-coated with 15 μm of platinum (Q300T D, UK) for 60 s at 30 mA. The microscopic images were further analysed at ×400, ×600 and ×1000 magnifications using a Hitachi TM4000 Plus Tabletop SEM.

Statistical analysis

The data were analysed using the Origin 2020b software. The flexural, compressive, microhardness, fracture toughness, and water sorption data were analysed using the one-way analyses of variance (ANOVA) and subjected to Tukey's *post-hoc* test. One-way ANOVA was used to evaluate if the groups had a measurable effect on the variable mentioned above, and Tukey's *post-hoc* test was used to determine significant differences between groups, where $P = 0.05$ was considered statistically significant. Additionally, a regression and correlation analysis were conducted to investigate if there is a correlation between the filler wt. % and the compression and flexural elastic moduli and surface microhardness.

RESULTS

Flexural strength and modulus

The flexural strength and elastic modulus results of the tested groups, along with standard deviations, are presented in Fig. 1. Ste_Cap had the highest mean flexural strength at 113.4 MPa, higher than all other groups tested in the study. No significant differences ($P < 0.05$) were noted between the mean flexural

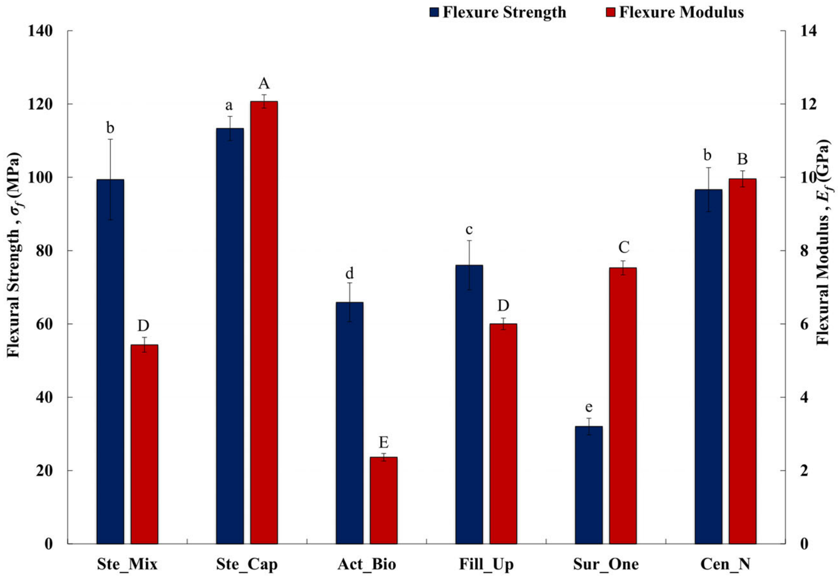

Fig. 1 The flexural strength and modulus between commercially available self-cured and dual-cured DRCs. Groups with the same alphabet are not significantly different from each other ($P > 0.05$).

strengths of the self-cured composite Ste_Mix and Cen_N with Tukey's *post-hoc* comparison. However, all the other dual-cured commercial DRCs showed significant differences from each other. The flexural strength of the novel self-cure DRCs Ste_Mix and Ste_Cap also differed significantly (Fig. 1). Compared to the other groups in the study, Sur_One had the lowest flexural strength, at 32.0 MPa. The mean flexural modulus varied from 2.36 GPa (Act_Bio) to 12.07 GPa (Ste_Cap). According to Tukey's *post-hoc* comparison, no significant difference was found between Ste_Mix and Fill_Up.

There were significant differences in the flexural modulus of elasticity among the commercial dual-cure DRCs, such as Act_Bio, Sur_One, and Cen_N, compared to the self-cure composite Ste_Cap. There was also a significant difference in the flexural modulus between the two novel self-cure DRCs Ste_Mix and Ste_Cap (Fig. 1). All tested samples showed a linear load-displacement behaviour and fractures before deforming plastically, which is a characteristic behaviour of brittle materials (as shown in the supplementary image S1). The analysis of the variance of the flexural strength and modulus is shown in S2. A positive correlation was found between the filler wt.% and the flexural modulus ($R^2 = 0.93$), as shown in Fig. 3. When preparing the correlation between filler weight and flexural modulus, the Sur_One was excluded because of its low value, possibly due to water in its composition. Therefore, this cannot be accounted for to understand the influence of filler weight and flexural modulus. The mathematical equation between the flexural modulus resulting from filler wt.% is presented in Eq (9):

$$\mathit{Flexural\ Modulus}\ E_f = 0.38\ (\mathit{Filler\ wt.\%}) - 18.20 \tag{9}$$

Compressive strength and modulus

The mean compressive strength and modulus values from the tested groups, along with the standard deviations, are shown in Fig. 2. The group with the highest mean compressive strength value was Fill_Up with 223.5 MPa, while the group with the lowest compressive strength was Sur_One with 119.3 MPa, as seen in Fig. 2. Based on Tukey's *post-hoc* comparison, a statistically significant similarity between self-cure DRCs Ste_Mix, Ste_Cap and the dual-cured dental composite Fill_Up and Cen_N. Similarly, a statistical similarity was seen between Ste_Mix and Act_Bio. However, there were significant differences in the compressive strength between Sur_One and the rest of the groups. The modulus of elasticity in compressive was also determined (Fig. 2).

The compressive modulus varied from 1.84 GPa (Sur_One) to 4.1 GPa (Ste_Cap). According to Tukey's *post-hoc* comparison, the elastic modulus values of the dual-cure DRCs significant similarity was noticed amongst Ste_Mix, Ste_Cap and Act_Bio, while the Fill_Up, Sur_One and Cen_N varied significantly between each other (Fig. 2). The analysis of the variance of the compression strength and modulus is shown in S2. A high positive correlation was found between the compressive elastic modulus and the filler wt.% ($R^2 = 0.90$), as shown in Fig. 3. Since Sur_One has a low value, it was excluded from the correlation between filler wt. % and compression modulus. This is possibly a consequence of the water content in its composition.

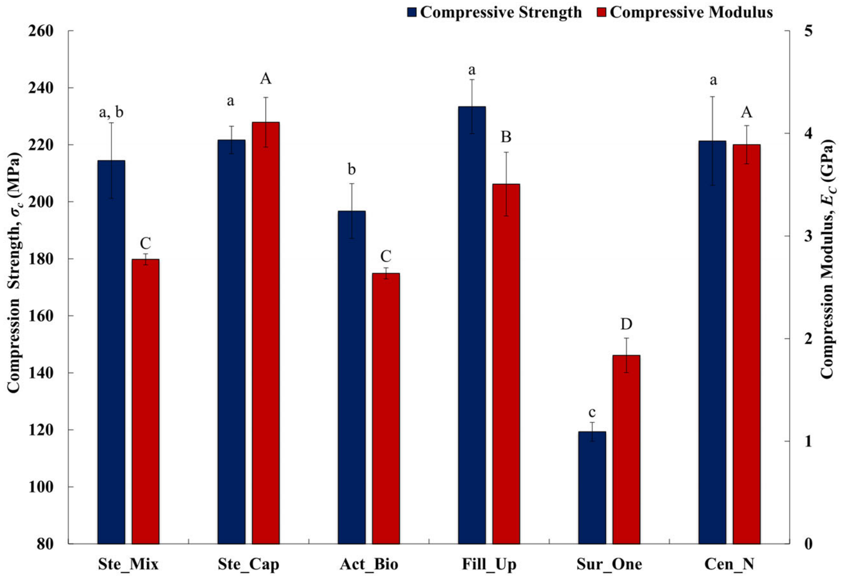

Fig. 2 The influence on the compressive strength and modulus between different commercially available self-cure and dual-cured DRCs is studied. Groups with the same alphabet are not significantly different from each other ($P > 0.05$).

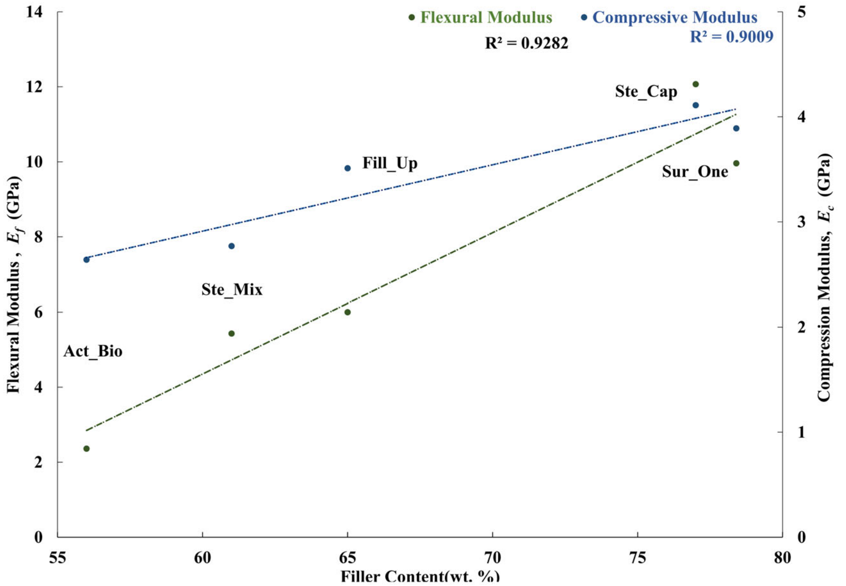

Fig. 3 A linear correlation between the moduli and filler content (wt.%) was observed with the R^2 value of 0.93 and 0.88 for flexural and compressive, respectively. The flexural and compressive moduli of Sur_One were not considered as they showed inferior elastic properties.

It is, therefore, irrelevant to understanding the relationship between filler weight and compression modulus. Equation (10) describes the relationship between compression modulus and filler wt. %.

$$Compression\ Modulus\ E_c = 0.06\ (Filler\ wt.\%) - 0.88 \quad (10)$$

Fracture toughness

Mean values for fracture toughness (K_{IC}) with standard deviations are shown in Fig. 4. The fracture toughness values were highest with the self-cure dental composite Ste_Cap at 1.41 MPa.m$^{0.5}$, while it was the lowest for the dual-cure dental composite Sur_One at 0.43 MPa.m$^{0.5}$. Tukey's *post-hoc* comparison ($P < 0.05$) showed statistically significant differences Ste_Mix, Ste_Cap, Fill_Up, Sur_One and Cen_N. Act_Bio was found to be statistically similar to Ste_Mix (Fig. 4). The analysis of the variance of the fracture toughness for different groups is shown in S2.

Surface microhardness

The surface microhardness of commercial self-cured DRCs and dual-cured DRCs was assessed, and the significant differences between the groups were measured. The results of the VH tests are shown in Fig. 5. It is visible that each dental composite group varied significantly from the others, except Sur_One and Cen_N, which had no significant differences ($P > 0.05$) between each other (Fig. 5). The self-cured and dual-cured composites had varying hardness levels, ranging from 24.1 N/mm^2 for Act_Bio to 68.3 N/mm^2 for Ste_Cap. The analysis of the variance of the surface microhardness for different groups is shown in S2. A positive correlation was found between the filler wt.% and the surface microhardness of the tested groups ($R^2 = 0.86$), as shown in Fig. 6. The mathematical equation between the surface microhardness resulting from filler wt.% is presented in Eq (11):

$$Surface\ microhardness\ (VH) = 1.56\ (Filler\ wt.\%) - 54.37 \quad (11)$$

Fractured surface morphology

The fracture surfaces of the tested groups were investigated using SEM analysis and are shown in Fig. 7. SEM evaluation presented a conventional structure of each investigated self- or dual-cured DRCs with different filler particulate shapes and sizes in the composite matrix.

Based on the SEM images (Fig. 7), it is evident that the filler particle sizes in the Sur_One, Ste_Mix, and Ste_Cap groups are smaller in comparison to those in the other groups, namely, Act_Bio, Fill_Up, and Cen_N. This indicates that the former composites demonstrated a more homogeneous submicron filler composition, integrating well into the surrounding resin matrix. The SEMs showed that Act_Bio, Fill_Up and Cen_N contained different fillers of varied sizes (Figs 7c, d and 7f). However, the SEM images showed that Ste_Cap showed fewer cracks and pits than Ste_Mix, indicating higher strength values in the former than in the latter (Figs 7a and 7b). Amongst the self-cured and dual-cured DRCs, Sur_One exhibited many cracks and pits (Fig. 7e) on the sample. This suggests

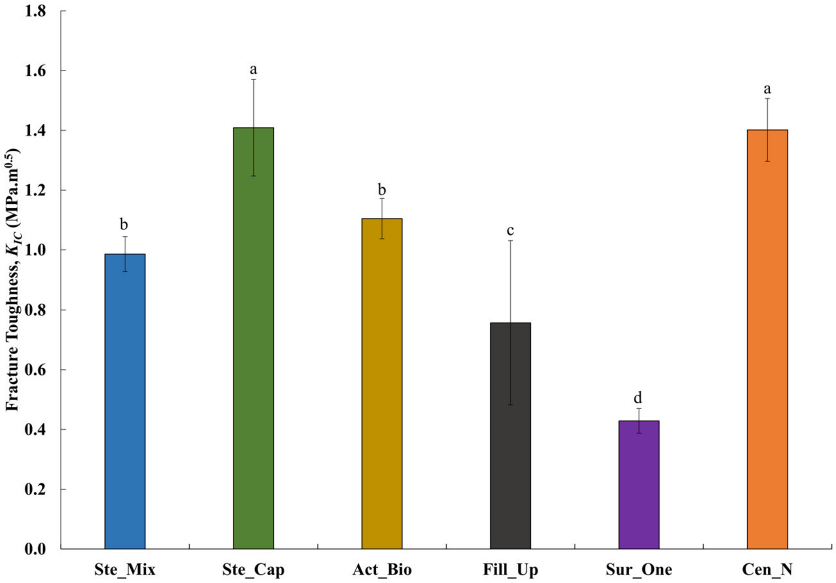

Fig. 4 Influence on the fracture toughness between different commercially available self-cure and dual-cured DRCs is analysed. Groups with the same alphabet are not significantly different from each other ($P > 0.05$).

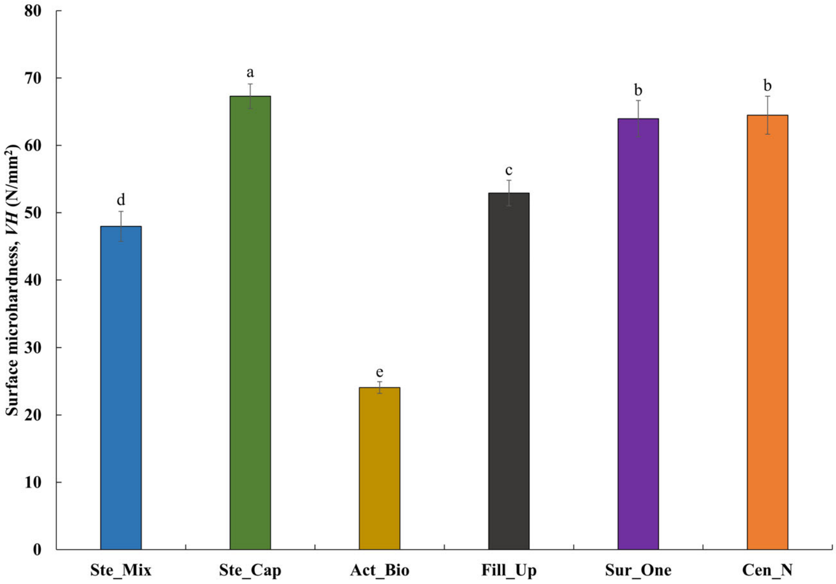

Fig. 5 The VH results are studied between commercially available self-cured and dual-cured DRCs. Groups with the same alphabet are not significantly different from each other ($P > 0.05$).

the loss of water from the sample, as water is one of the main components in Sur_One (Table 1). Therefore, Sur_One showed extremely low fracture toughness values and flexural and compressive strength compared to commercial DRCs. It was also noticed that surface cracks were formed on the fillers of Fill_Up than on the resin (Fig. 7d). Additionally, the micro-sized fillers on Act_Bio were surrounded by pits, which could indicate the low interface strength between the resin and the fillers. These findings indicated an explanation for the observed differences in the mechanical and physical behaviours of the DRCs.

Water sorption and solubility properties

The mean water sorption and solubility values from the study are shown in Fig. 8. The water sorption values range from 158.4 ± 1.6 μg/mm^3 for Sur_One to 13.8 ± 0.6 μg/mm^3 for Ste_Cap. The mean water solubility values range from 1.8 ± 0.3 μg/mm^3 for Ste_Cap to 22.7 ± 0.2 μg/mm^3 for Sur_One. Tukey's test showed that there is a significant difference among the mean values of the groups while comparing the water sorption results ($P < 0.05$). However, this is not true in solubility results, as there is significant

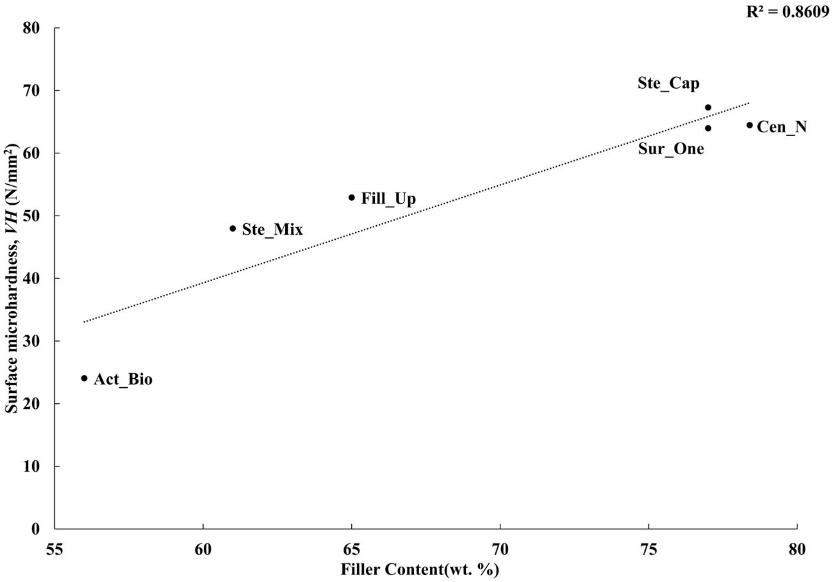

Fig. 6 The VH results are based on the filler content of the self- and dual-cured composites. A linear correlation between microhardness (VH) and filler content (wt.%) was observed with the R^2 value of 0.86.

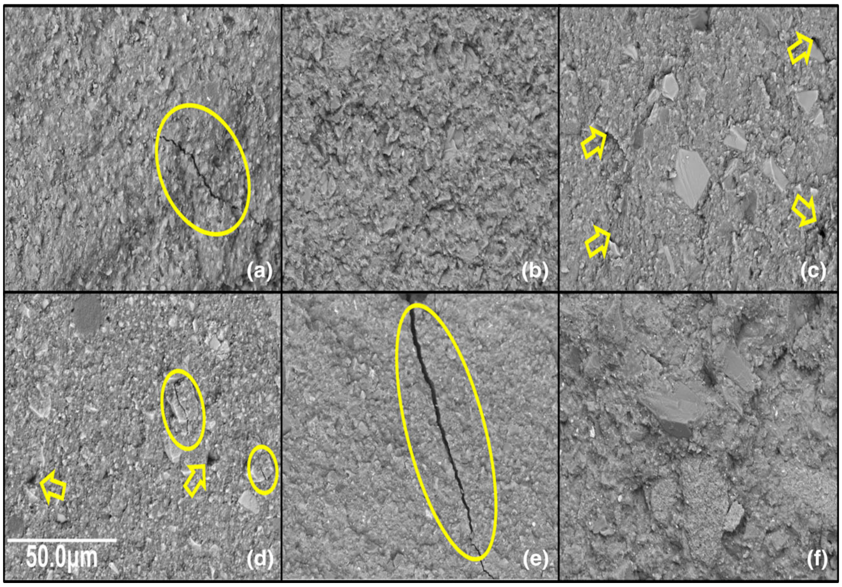

Fig. 7 SEM photomicrographs (scale: 50.0 µm, magnifications: ×1000) of investigated self-cured and dual-cured DRCs after the fracture toughness testing. (a) Ste_Mix; (b) Ste_Cap; (c) Act_Bio; (d) Fill_Up; (e) Sur_One; (f) Cen_N. The arrows indicate small pit defects, and the circular markings indicate the crack formed.

similarity between the mean W_{sl} values of Fill_Up and Cen_N. According to the ISO 4049 standard,[20] the W_{sp} and W_{sl} values must be lower than 40 µg/mm^3 and 7.5 µg/mm^3, respectively. The W_{sp} and W_{sl} values obtained in this study were lower than the threshold values for all groups except Sur_One, which were much higher than the threshold values. The analysis of the variance of the water sorption and solubility properties for different groups is shown in S2.

DISCUSSION

The studies were conducted to analyse the mechanical and physical properties of commercially available dual-cured DRCs and self-cured DRCs. Within the study's limitations, which depend on the data provided by the manufacturers for the commercially available dual-cured DRCs, and considering that filler and matrix phases have different compositions and

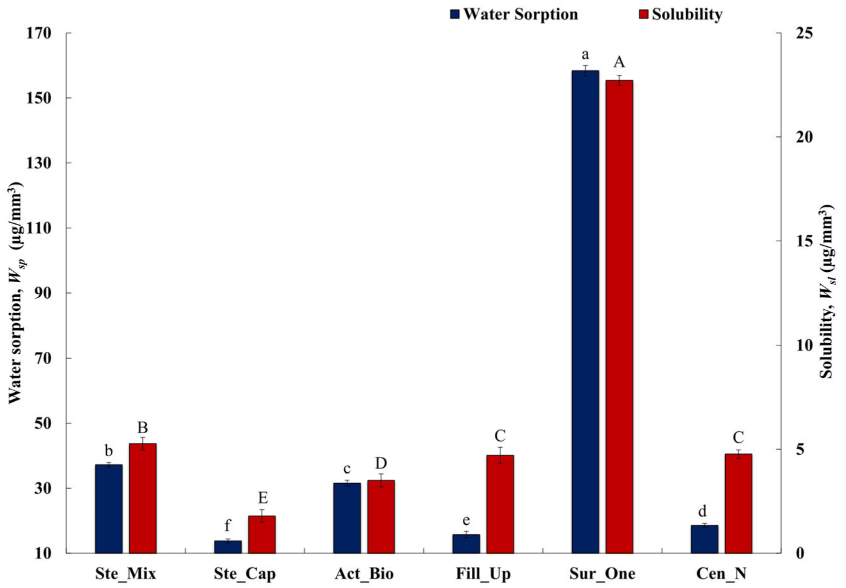

Fig. 8 The influence on the water sorption and solubility between self-cured DRCs and dual-cured DRCs grafted are studied. Groups with the same alphabet are not significantly different from each other ($P > 0.05$).

chemistries for each product examined, the results have shown that the mechanical and physical properties of the tested self- and dual-cured DRCs are different.

Parameters influencing the flexural and compressive modulus of DRCs.

The elastic modulus from the flexural and compressive tests provides insight into the materials' rigidity.[29] A material with a higher elastic modulus has been shown to require more stress for the same degree of deformation,[30,31] thereby giving a more rigid property. The results from this study showed that there is a strong correlation between the elastic modulus and the filler content wt. % (Fig. 3). Although higher values of elastic modulus would increase the rigidity of the material, a study has shown that it also increases the shrinkage stress within the dental composite.[32] This could eventually lead to higher deformation and fracture in the composite.[31] However, the exceptionally low values of the elastic modulus of Sur_One contravene the suggested linear correlation of filler wt.% – elastic modulus. This may be due to the significant amount of water in the composite composition (Table 1), which causes a lower degree of resin-filler adhesion due to the degradation of the resin from the surrounding filler particles.[33,34]

Parameters influencing the flexural and compressive strength of DRCs.

Studies[35–37] have shown that flexural and compressive strength increases with the increase in filler loading. However, in this study, there was no linear correlation between the strength and filler loading of the composite. It was seen, in previous studies,[36,38] that for DRCs with resin monomers such as bis-GMA, UDMA and TEGDMA, the flexural strength of the material ranked in the order of UDMA followed by TEGDMA and finally bis-GMA. This may be due to the degree of conversion of the polymer matrix. An increase in flexural strength was noticed with the increase in the degree of conversion, which is higher for more flexible monomers seen in UDMA and TEGDMA.[38,39] This is possibly the reason for the lower strength of Act_Bio compared to the other DRCs chosen for the study. Another reason Act_Bio and Sur_One showed low mechanical properties may be due to the competition between the acid-base reaction and the resinous polymerization reaction that occurs during the curing.[40,41] This competition in reactions could explain the lower mechanical properties[22,41] when compared to dental materials such as Cen_N, which are not based on dual-setting reactions. The self-cured DRCs (Ste_Mix and Ste_Cap) showed the highest mechanical strength. This may be due to the high cross-linking ability of GDMA[42] present in Ste_Mix and Ste_Cap. This helps in the easier cross-linking of the resin matrix with the fillers, thereby increasing the mechanical properties compared to the dual-cured DRCs. The higher to average flexural and compressive strengths of Cention N could be linked to the substantial mechanical properties and long-term stability that can be attributed to the combination of UDMA, DCP, and an aromatic aliphatic-UDMA and PEG-400 DMA, which interconnects (cross-links) during polymerization.[43]

Parameters influencing the surface hardness of DRCs.

The VH test method is a commonly used test technique in determining the surface microhardness of the composite. According to ISO standards, the dental restoration composite material should have a minimum VH value of 40 HV.[44] The hardness values for the groups chosen for this study are all above the required standard value of 40 HV, except Act_Bio, which is less than 40 HV. Studies have shown that microhardness values of a composite can be affected by several factors such as organic and inorganic composition, filler content and the distribution on the surface, and degree of C=C conversion in methacrylate groups and cross-linking density.[45,46] In this study, a linear correlation ($R^2 = 0.86$) can be observed between the filler content (wt.%) and the microhardness (Fig. 6). This increase in the microhardness values with respect to the filler content agrees with other previous studies.[47–50] However, as stated above, the filler content may not be the only primary factor affecting the microhardness properties of a composite. The probable reason for a high microhardness value in Ste_Cap compared to Sur_One, even though the filler wt.% is the same, may be due to the variation in the organic resin matrix used.[47] The high microhardness value of Cen_N may be due to a nanoparticle-sized patented filler called 'isofiller'.[43] However, the microhardness values from this study are comparatively low when compared to other literature. This is because the microhardness values also correlate with the degree of monomer conversion in composites. The degree of conversion is expected to be low at the surface due to surface oxidation and the rapid migration of radicals into the material, resulting in oxygen inhibition of the surface layer.[51]

Parameters influencing the fracture toughness of DRCs

Prior studies have identified a positive linear correlation between the stress intensity factor (K_{IC}) and the filler load wt.%,[52–54] while few studies have shown that the K_{IC} values decline when the filler load exceeds 55 vol.%.[52] However, this study did not see a positive correlation between the filler load wt.% and the KIC, suggesting other factors affecting the K_{IC}. Studies have shown that factors such as the shape, size, and distribution of filler particles also influence the fracture toughness of the composite.[52] Moreover, if the adhesion between the filler and matrix is not strong and if any voids appear between the filler and the resin matrix, these voids may act as initial fracture sites (stress concentrators) in the composite and facilitate the breakdown of the material.[52] High values of K_{IC} were seen for both Ste_Cap and Cen_N, followed by Act_Bio. The increased values in Ste_Cap and Act_Bio, which are nano-composites, may be due to the reduced flaw/defect density, reduced flaw/defect size, or an increase in monomer conversion. The values of K_{IC} can also be seen increasing in nano-composites as they have enhanced bonding at the filler-matrix interface, higher strength fillers, and an increased surface area-to-volume ratio.[52] The high K_{IC} of Cen_N could also be due to the high filler contents of barium aluminium silicate glass and calcium aluminium silicate glass.[55] The average KIC value for Fill_Up was significantly lower than that of all other materials, except for Sur_One. This may be due to the effects of particle interferences and the lower modulus of elasticity found in microfilled urethane composites. A reduction in KIC values can also be caused by air voids, interfacial flaws, broken fillers, filler agglomerates, and a reduced polymerization of monomers in highly packed composites.[58] Studies have also shown that UDMA in the resin has a higher level of water absorption behaviour compared to other bis-GMA or TEGDMA-based composites. While slight hydrolytic plasticization can improve a material's fracture toughness, excess plasticization reduces its fracture toughness properties. Although slight hydrolytic plasticization may improve a material's fracture toughness, excess plasticization reduces its fracture toughness properties.[56,57]

Parameters influencing the fractured surface morphology of DRCs.

The SEMs at ×1000 magnifications were obtained for all the groups (self- and dual-cured DRCs). The SEM photomicrographs (Fig. 7) of the groups displayed smooth surfaces and did not show any dark regions, indicating two significant observations. First, it suggests that during fracture toughness testing, a regular fracture occurred in the stressed area without loss of filler and organic particles.[59] Second, the absence of dark regions could also suggest that fillers have effectively deflected crack propagation. This indicates that more energy consumption is required during the break.[60] However, the presence of fillers cannot be the only reason for the increase in fracture toughness properties. Moreover, studies[61,62] have shown that adding fillers sometimes may decrease the mechanical strength of the composite. This could be seen in Fill_Up, where the composites have cracks through the fillers. During the testing, the loads are transferred onto the surface of the particles through the resin matrix. Since the particles were harder than the resin matrix in which they were embedded, much of the stress was transferred through the particle and back into the resin itself. Wherever the filler particles were of irregular shapes or angulated, the stress concentrations became excessively high. This would therefore create small cracks around the particles, thereby weakening the matrix locally.[61] Except for Fill_Up and Sur_One, the SEMs of all the other samples show that the filler particles had higher adhesion with the matrix. This suggests the fillers could deflect crack propagation, indicating good filler/matrix adhesion.[33,54,62] Good filler/matrix interfacial adhesion indicates enhanced strength and fracture energy.[62–65] For the Sur_One, crack path observations showed clear interfacial separation between the filler particles and matrix. Studies[33,34,66,67] have shown that hydrated composites (water being a composition ingredient) had a lower degree of adhesion between the resin and filler, probably due to degradation of the resin matrix or the silane layer surrounding the filler particles from the excess amount of water. This could be the reason for low fracture toughness behaviour of Sur_One.

Parameters influencing the hydrolytic degradations of DRCs

Analysing the water sorption (W_{sp}) and solubility properties is essential, as the diffusion of water molecules that occurs within the matrix of the DRCs when saliva is in contact with the tooth can affect and compromise the physical and mechanical properties of the dental restorations.[36,68] Depending on the microstructural and molecular aspects, the polymers tend to absorb different amounts of water. The polarity of the molecular structure, the presence of hydroxyl groups capable of forming hydrogen bonds with water, and the degree of cross-linking in the continuous matrix influence the absorption and solubility of water.[69] The absorption of water may lead to the formation of gaps between polymer chains. This gap allows unreacted components trapped within the polymer chains to diffuse out, leading to increased solubility.[69,70] When the type of storage medium and the immersion period of DRCs are kept constant, other factors affecting the solubility are the surface area of the filler used, the particular silane treatment, the difference in the monomer structure, and the degree of cross-linking.[71]

Previous studies show that Wsp values negatively correlate with the amount of filler content.[69,72] Studies have suggested this is because the interfacial gaps between the matrix and the filler particles can accommodate absorbed water.[73] The results from our study have shown partial agreement with the above statement, although Act_Bio, which has the lowest filler content (56 wt.%), did not show the highest values of water sorption and solubility. However, Ste_Mix, which had a higher filler ratio (61 wt.%) than Act_Bio, showed the highest water sorption and solubility values. This can also be seen for Cen_N, which has the highest filler content (78.4 wt.%), yet it shows higher water sorption and solubility when compared to Ste_Cap which has lower filler wt.% than Cen_N. This indicates that the water sorption and solubility properties depend not solely on the filler content. Studies[74,75] have shown that dual-cured DRCs are more hydrophobic than self-cured DRCs. Therefore, Cen_N showed higher water sorption and solubility when compared to Ste_Cap. Another reason for Cen_N to show higher water sorption and solubility when compared to Ste_Cap is the presence of UDMA in Ste_Cap (Table 1). UDMA is more hydrophobic than hydrophilic BisGMA, GDMA, HEMA or TEGDMA, absorbing less water.[42,75] Sur_One showed the highest water sorption and solubility properties compared to all other groups and exceeded the permitted standards of 40 μg/mm^3 and 7.5 μg/mm^3, respectively. This nature of very elevated water sorption and solubility may be due to the composition and chemical reactions. Sur_One, which is partly composed of water, theoretically promotes water and ion exchange with the oral environment.[40] This effect also leads to the release of fluoride, aluminium, and calcium ions (and probably other ions due to the composition of the reactive fillers),[40] increasing its solubility and water sorption values. Studies[76,77] have shown that composites that contained Zinc, Barium and Strontium glass leached more into the water when compared to composites that incorporated particles of silica. This could be the cause of the higher solubility value for Ste_Mix when compared to Fill_Up. Another reason for the increase in water sorption and solubility values may be due to voids in the composites formed due to the large filler surface area.[78]

CONCLUSION

This study focuses on understanding the physical and mechanical behaviour of different restorative materials with varying constituent compositions. Despite the limitations of not conducting actual measurements of monomer content, the following conclusions can be drawn based on manufacturer data:

1. There is a strong correlation between elastic moduli (flexural and compressive) and filler content, except Sur_One, where water disrupts resin-filler adhesion.
2. The resin type plays a crucial role in flexural and compressive strength, with GDMA and TEGDMA-based composites demonstrating superior conversion and cross-linking capabilities.
3. Filler shape, size, and distribution affected fracture toughness, with nano-composites

demonstrating enhanced toughness due to improved bonding between filler and matrix.

(4) Analysing fractured surface morphology reveals that fillers deflect crack propagation, and adhesion between fillers and matrix is critical to improving toughness.

(5) Filler content and resin matrix influenced surface hardness, with Cention N showing high hardness due to nanoparticle-sized fillers (isofiller).

(6) A linear relationship is identified between the filler content (wt.%) and the surface microhardness (HV).

(7) Flexural and compression moduli and surface microhardness of a DRC can be predicted using the regression analysis of the filler wt.% data.

(8) Hydrolytic degradation assessment highlights that filler content, resin, and cross-linking degree influence water sorption and solubility.

(9) Sur_One is highly hydrolytically unstable due to its significant water content, leading to ion exchange with the oral environment.

Overall, the variance in results from the study emphasises careful consideration when selecting materials for specific clinical applications and can lead to enhanced dental restorations with improved longevity and performance.

ACKNOWLEDGEMENT

This research was supported by the AusIndustry Cooperative Research Centre Project (CRC-P), Grant CRCPSEVEN000013, which is gratefully acknowledged by the authors. The authors are thankful to Dr Chee Wah Loy, School of Mechanical and Manufacturing Engineering at UNSW, for support in the microhardness tests. The authors would like to acknowledge the facilities and technical assistance of Microscopy Australia at the Electron Microscope Unit (EMU) within the Mark Wainwright Analytical Centre at UNSW Sydney. The authors also acknowledge the support received from the ARC Training Centre for Automated Manufacture of Advanced Composites (IC160100040), supported by the Commonwealth of Australia under the Australian Research Council's Industrial Transformation Research Program. Open access publishing facilitated by University of New South Wales, as part of the Wiley - University of New South Wales agreement via the Council of Australian University Librarians.

SUPPORTING INFORMATION

Additional Supporting Information may be found in the online version of this article:

Data S1.

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Address for correspondence
J Thadathil Varghese and B Gangadhara Prusty
School of Mechanical and Manufacturing Engineering
University of New South Wales
Sydney
NSW
2052,
Australia
Email: j.thadathil_varghese@unsw.edu.au;
g.prusty@unsw.edu.au