

Effect of Dietary Challenges on the Flexural Properties of Resin-based Composites

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KEYWORDS

Resin-based composites, food-simulating liquids, dietary solvents, flexural strength, flexural modulus

ABSTRACT

This study aims to compare flexural strength and flexural modulus of different resin-based composites (RBCs) and to determine the impact of dietary solvents on flexural properties. Forty specimens (12x2x2mm) for each of two conventional (Aura Easy [AE]; Harmonize [HN]) and one bulk fill (Sonic Fill 2 [SF2]) were fabricated using customised plastic moulds. Specimens were light-cured, measured and randomly divided into four groups. The groups (n=10) were conditioned for 7 days at 37°C: in one of media: air (control), artificial saliva (SAGF), 0.02N citric acid and 50% ethanol–water solution. After conditioning, the specimens subjected to flexural testing. Two-way ANOVA and one-way ANOVA (post hoc: Tukey's or Dunnett T3 tests) were used at $\alpha=0.05$. Significant differences in flexural properties were observed between materials and conditioning media. Flexural strength and modulus values ranged from 124.85MPa to 51.25MPa; and 6.76GPa to 4.03GPa, respectively. The highest flexural properties were obtained with conditioning in air. Exposure to aqueous solutions generally reduced flexural properties. In conclusion, the effect of dietary solvents on flexural properties were material and medium dependent. For functional longevity of restorations, patients' alcohol intake should be considered during material selection. Dietary advice (reduce alcohol consumption) should be given to patients post operatively.

INTRODUCTION

In modern dentistry today, increased aesthetic and mechanical demands for restorative materials to mimic natural tooth foster the development and consistent improvements of direct tooth coloured restorative materials [1]. Materials available range from resin-based composites (RBCs) to glass ionomer cements with hybrid ionomers in between [2]. RBCs have evolved significantly in dental practice since their introduction 50 years ago. Modification of several factors such as chemical composition, filler content and size of filler particles aimed to improve their clinical handling and

performance [2]. Consequently, new RBCs are constantly introduced by different manufacturers into the dental market.

RBCs are routinely subjected to certain amounts of flexural forces and stresses from masticatory actions in the oral environment, which have considerable effects on their durability and clinical performance [3]. Therefore, flexural properties are crucial factors in selecting dental restorative materials for the clinical durability of restoration [4]. In view of this, it is important for dental clinicians and material researchers to identify the flexural properties (flexural strength and flexural modulus) of RBC materials [5]. Flexural strength, which measures the ability of a material to bend before breaking, is one of the key values that represents the longevity and durability of dental materials [6]. Thus, dental restorative materials are formulated to meet the criteria of high flexural

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strength because they are subjected to adverse oral environment and considerable masticatory force, which cause permanent deformation [6]. On the other hand, flexural modulus indicates the stiffness of dental materials. Materials with higher flexural modulus are stiffer than materials with lower flexural modulus [7].

Besides intrinsic properties of materials, long term clinical success of dental restorative materials is also determined by their properties after exposure to the oral environment [4]. Restorative materials are constantly exposed to the harsh environment in the oral cavity due to the chemical attacks from saliva, food products, temperature changes and fluctuations in pH [8,9]. Adverse oral environment challenged the physical and mechanical properties of restorative materials under clinical conditions [10]. These can cause chemical degradation of RBC restorations involving the softening of resin matrix, hydrolytic degradation of fillers and silane couplers as well as leaching and debonding of fillers [11]. Food simulating liquids (FSL) [12] are dietary solvents which can simulate conditions in oral cavity that allows the evaluation of dental restorative materials in a brief period of time and also consider chemical affinity, elution and bonding process [13]. Artificial saliva is used to mimic natural saliva while citric acid and ethanol are used to imitate some fruits and vegetables, sweets and alcohol drinks [13,14,15].

To date, there are limited studies on flexural properties as well as the impact of dietary solvents on their flexural properties [16,17] of recently developed RBCs namely Aura Easy, Harmonize and Sonic Fill 2. Aura Easy contains ultra-high density glass fillers whose unique morphology can improve the mechanical properties of the composite. Adaptive Response Technology (ART), adopted by Harmonize, involves the fusion of zirconia and silica nanoparticles forming a stabilised filler network for enhanced mechanical strength [18]. Sonic Fill 2 is an upgraded version of Sonic Fill 1, which presents better mechanical properties due to the employment of new filler system composed of zirconium oxide and silica oxides nanoparticles, while preserving the sonic-activation technology [19]. Therefore, this research aimed to compare the flexural strength and flexural modulus of various RBCs and to investigate the impact of dietary solvents on their flexural properties. The null hypotheses were as follows: (a) the flexural properties between different RBCs have no significant difference and (b) conditioning media do not significantly influence various RBCs' flexural properties.

MATERIALS AND METHODS

A laboratory study was carried out. Three light cured RBCs were evaluated in this study. These are Aura Easy (SDI, Bayswater, Victoria, Australia), Harmonize (Kerr, Orange, CA, USA) and Sonic fill 2 (Kerr, Orange, CA, USA). Technical profiles and manufacturers of these RBCs are presented in Table 1.

Specimen preparation

Customised plastic moulds were used to fabricate a total of forty beam-shaped test specimens (12mm x 2mm x 2mm) for each of the tested materials. The materials were sonic activated using the handpiece following manufacturers' instructions (where applicable) and/or placed into the moulds in a single increment. Both ends of the filled moulds were compressed using a mylar strip and glass slide to remove excess material.

Specimens were light cured from the top surface through the glass slide with two overlapping irradiations of 10s or 20s each using a calibrated LED curing light (Demi Plus, Kerr, CA, USA) with an output irradiance of 1330 mW/cm² and the wavelength range was between 450-470nm. Glass slides were removed and the specimens were light cured for another 10s. The bottom surface was then light cured for another 10s or 20s. The mylar strips were removed and the beam shaped specimens were separated from their moulds. The specimens were visually examined for the presence of voids. Specimens with defects were replaced. Digital calliper (Mitutoyo Corporation, Kawasaki, Japan) was used to verify the dimensions of the specimens and parallelism of its opposite surfaces.

Conditioning media and time

The measured specimens were randomly allocated into four groups of ten (n=10) and were conditioned in the following media in closed containers for 7 days at 37°C: air (control), artificial saliva (SAGF), 0.02N citric acid and 50% ethanol-water solution [12]. Table 2 shows the constitution of artificial saliva [20]. Containers filled with the test media were sealed to reduce evaporation and air exposure. They were stored in an incubator at 37°C (Mettler, IN-460, Schwabach, Germany). The pH of the artificial saliva was verified using a digital pH meter (Eutech pH2700, Singapore) and was adjusted to 6.8 with diluted hydrochloric acid (when needed) to simulate the pH of natural saliva when it is released from the salivary ducts.

Table 1 Technical profiles and manufacturers of the materials evaluated

Materials	Manufacturer	Classification	Resin	Fillers types	Filler size (nm)	Filler content % by weight/volume
Aura Easy (AE)	SDI, Bayswater, Victoria, Australia	Conventional nanohybrid composite	Bis-EMA, UDMA, TEGDMA	Barium aluminoborosilicate glass, Silica	20-400	81/65
Harmonize (HN)	Kerr Corporation, Orange, CA, USA	Conventional nanohybrid composite	Bis-GMA, TEGDMA, Bis-EMA	Silica, zirconia, barium glass	5-400	81/64.5
SonicFill 2 (SF2)	Kerr Corporation, Orange, CA, USA	Sonic-activated bulk-fill nanohybrid composite	Bis-GMA, TEGDMA, Bis-EMA	Silicon dioxide, barium aluminoborosilicate glass, Ytterbium trifluoride	40-800	83.5/66.5

Bis-EMA= Ethoxylated bisphenol-A-glycidyl methacrylate; Bis-GMA=Bisphenol-A glycidyl methacrylate; TEGDMA=Triethylene glycol dimethacrylate; UDMA=Diurethane dimethacrylate
*(Abbreviation) depicts the code for study materials

Table 2 Composition of the SAGF media (Gal et al., 2001)

Components	Concentration (mg L-1)
NaCl	125.6
KCl	963.9
KSCN	189.2
KH ₂ PO ₄	654.5
Urea	200.0
NaSO ₄ •10H ₂ O	763.2
NH ₄ Cl	178.0
CaCl ₂ •2H ₂ O	227.8
NaHCO ₃	630.8

Flexural Testing

After the 7-day conditioning period, the specimens were loaded until fracture using a universal testing machine (UTM) (Shimadzu Corporation, Kyoto, Japan) with a load cell of 5KN and crosshead speed of 0.5mm/min. Flexural strength, σ , in Megapascal (MPa) was calculated using the following equation:

$$\sigma = \frac{3PL}{2BH^2}$$

where P is the maximum load exerted on the specimen in Newton, L is the distance between the supports in millimetres (10mm), B is the width of the specimen in millimetres, and H is the height of the specimen in millimetres.

Flexural modulus, E', in Megapascal (MPa) was calculated using the following equation:

$$E' = \left(\frac{F}{D}\right) \left(\frac{L^3}{4BH^3}\right)$$

where F/D is the slope, in newton per millimetre, measured in the straight-line portion of the load-deflection graph. L, B and H had been defined in the flexural strength equation. Flexural modulus was subsequently converted to Gigapascal (GPa).

Statistical analysis

SPSS (Version 12.0.1, SPSS Inc., Chicago, USA) was used to analyse the data. Data was checked for normality using the Shapiro-Wilk test. The data was found to be normally distributed. Thus, parametric analyses were performed. The interactions between the independent variables (materials and conditioning media) and each of the dependent variables (flexural strength and flexural modulus) were evaluated using two-way ANOVA. One-way ANOVA followed by Tukey's or Dunnett T3 post-hoc test was used to determine inter-medium and inter-material differences for both flexural strength and modulus. All statistical analyses were carried out at a significance level of $\alpha = 0.05$.

RESULTS

Mean flexural strength and modulus with their respective standard deviation (SD) values for RBCs evaluated after conditioning in the various media are shown in Table 3. The mean flexural strength and modulus for each material in the different media are shown in Figures 1 and 2. Statistical inter-material and inter-medium comparisons of mean flexural properties are shown in Table 4 and Table 5. Two-way ANOVA presented significant interactions between materials and media for flexural strength ($p < 0.001$) and flexural modulus ($p = 0.023$). Flexural properties of the materials were dependent on materials and conditioning media.

Flexural strength

Comparison between media

Conditioning in air resulted in highest flexural strength. Conversely, the lowest flexural strength was noted after storing in ethanol except for SF2. For SF2, lowest flexural strength was obtained in artificial saliva, however, there was no significant difference in flexural strength between artificial

saliva, citric acid and ethanol. All materials conditioned in air presented significantly larger value of flexural strength compared to those in aqueous solutions. HN and AE had a significantly lower flexural strength after conditioning in ethanol compared with other media.

Comparison between materials

Flexural strength of AE was highest among the materials tested in all media except ethanol (Fig 2). For ethanol, SF2 was observed to have the greatest flexural strength. RBC that presented the lowest flexural strength when conditioned in artificial saliva and ethanol was HN. However, SF2 presented the lowest flexural strength in air and citric acid. There was no significant difference in flexural strength between materials in citric acid. When conditioned in air, AE presented significantly greater flexural strength compared to SF2, whereas in ethanol, flexural strength of SF2 was significantly greater than AE. Both SF2 and AE had significantly greater flexural strength than HN in artificial saliva and ethanol.

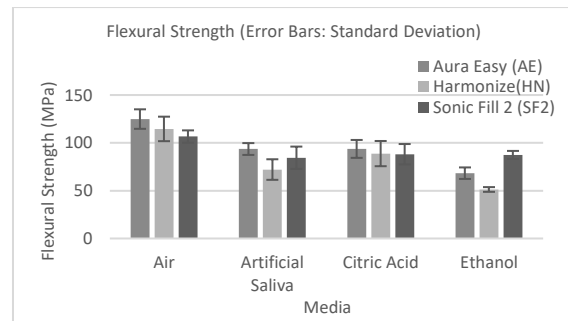


Figure 1 Mean flexural strength values (MPa) with standard deviation after storage in the different conditioning media

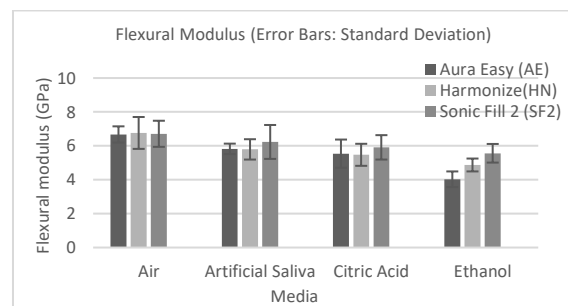


Figure 2 Mean flexural modulus values E' (GPa) with standard deviation after storage in the different conditioning media

Table 3 Mean flexural strength (MPa) and flexural modulus (GPa) of the various RBCs (standard deviations in parentheses)

Medium/ Material	Flexural Strength (MPa)				Flexural Modulus (GPa)			
	Air (control)	Artificial saliva	Citric Acid	Ethanol	Air (control)	Artificial saliva	Citric Acid	Ethanol
Aura Easy (AE)	124.85 (10.19)	93.56 (6.17)	93.68 (9.36)	68.29 (6.00)	6.67 (0.48)	5.82 (0.31)	5.54 (0.83)	4.03 (0.46)
Harmonize (HN)	114.56 (12.81)	72.08 (10.75)	88.76 (13.18)	51.25 (2.47)	6.76 (0.94)	5.79 (0.60)	5.47 (0.65)	4.87 (0.38)
Sonic Fill 2 (SF2)	106.60 (6.43)	84.45 (11.64)	88.10 (10.65)	87.35 (4.23)	6.71 (0.77)	6.23 (1.00)	5.91 (0.72)	5.56 (0.55)

Flexural modulus

Comparison between media

Flexural modulus of all materials tested showed the least decrease in air, followed in ascending order by artificial saliva, citric acid and ethanol. Conditioned in air resulted in the greatest flexural modulus, while exposure to ethanol resulted in the lowest flexural modulus (Fig 1). SF2 showed significantly higher flexural modulus when stored in air than in ethanol. The flexural modulus of HN conditioned in air and artificial saliva were significantly greater than in ethanol. AE, conditioned in air, showed significantly higher flexural modulus compared to those conditioned in the solutions, while significantly lower flexural modulus was shown after storing in ethanol compared to those in other media.

Comparison between materials

In artificial saliva, citric acid and ethanol, SF2 showed the highest flexural modulus. In air, HN had the greatest value of flexural modulus. In contrast, HN conditioned in artificial saliva and citric acid presented the lowest flexural modulus. For air and ethanol, AE showed the lowest flexural modulus. There was no significant difference in flexural modulus between restorative materials stored in air, artificial saliva and citric acid. After immersing in ethanol, SF2 had a significantly higher flexural modulus than AE and HN, while the flexural modulus of HN was significantly greater compared to AE.

Table 4 Comparison of mean flexural strength and modulus between different RBCs based on various media

Flexural properties	Mediums	p-value One Way Anova	Statistical Comparison between Materials	p-value post hoc test		
Flexural strength	Air	p=0.002	AE > SF2	p=0.001		
	Artificial saliva	p<0.001	SF2, AE > HN	SF2>HN	p=0.023	
				AE>HN	p<0.001	
	Citric acid	p=0.742	No significant difference			
	Ethanol 50%	p<0.001	SF2 > AE > HN	SF2>AE	p<0.001	
SF2>HN				p<0.001		
AE>HN				p<0.001		
Flexural modulus	Air	p=0.961	No significant difference			
	Artificial saliva	p=0.310	No significant difference			
	Citric acid	p=0.360	No significant difference			
	Ethanol 50%	p<0.001	SF2 > HN > AE	SF2>HN	p=0.008	
				SF2>AE	p<0.001	
HN>AE				p=0.001		

Abbreviations: AE, Aura Easy; HN, Harmonize; SF2, Sonic Fill 2

^aResults of One-way ANOVA and Post Hoc's Tukey's or Dunnett T3 test (p<0.05); > indicates statistical significance.

Table 5 Comparison of mean flexural strength and modulus between different media based on RBCs

Flexural properties	Materials	Statistical Comparison between Mediums
Flexural strength	AE	Air > Artificial Saliva, Citric Acid > Ethanol
	HN	Air > Citric Acid > Artificial Saliva > Ethanol
	SF2	Air > Artificial Saliva, Citric Acid, Ethanol
Flexural modulus	AE	Air > Artificial Saliva, Citric Acid > Ethanol
	HN	Air > Citric Acid, Ethanol; Artificial Saliva > Ethanol
	SF2	Air > Ethanol

Abbreviations: AE, Aura Easy; HN, Harmonize; SF2, Sonic Fill 2

^aResults of One-way ANOVA and Post Hoc's Tukey's or Dunnett T3 test (p<0.05); > indicates statistical significance.

DISCUSSION

The present research investigated the differences in flexural strength and flexural modulus of various RBCs and the impact of dietary solvents or FSL on their flexural properties. The flexural properties of RBCs were significantly influenced by both materials and conditioning media; thus, the null hypotheses were rejected.

The present study used the mini flexural test specimens (12mm x 2mm x 2mm) which have the advantages of reduced material usage and ease of specimens' fabrication, besides being more clinically realistic than ISO flexural test specimens (25mm x 2mm x 2mm) [5]. The elongated length of ISO 4049 flexural specimens is less clinically

appropriate due to the two factors mentioned below. First, it poses a technical challenge in fabrication due to the need to use multiple overlapping light irradiation because of the smaller light exit windows of most curing tips compared to the length of the specimens [21]. Next, the mesio-distal width of molars is, on the average, approximately 11mm and the cervico-incisal height of central incisors is approximately 13mm making its use less clinically relevant [22].

RBCs are predisposed to various complex intra-oral forces such as compressive, tensile and shear stresses during mastication, which challenge the mechanical strength of RBCs [3]. Masticatory pressure could be concentrated at a single point between opposing teeth or restorative materials after the food is ground and become alimentary bolus [23,24]. The concentration of stresses contributes to fracture of restorative materials. Therefore, in this study, static three-point bending test was employed to mimic mechanical stress in the oral cavity during mastication [24] and to determine the flexural properties of the RBCs. In contrast to the unwanted inertial effects of materials tested in dynamic test, static three-point bending test may be a helpful predictor of clinical performance and allows comparison of mechanical properties of materials being tested under a controlled situation [3,24]. Moreover, static test is carried out without the influences of other factors such as different temperatures and frequencies, thus, the isolated impact of dietary challenges on the deterioration of the tested materials could be clearly observed.

Although static test to evaluate flexural properties of composites is widely employed, the evaluation

by static test after immersion in FSL is a novelty. Chemical degradation of dental composites after exposure to adverse oral environment especially dietary liquids can remarkably challenge their flexural properties [25]. This is initiated by water absorption that causes dissolution or elution of leachable inorganic filler particles and residual monomers, thus destroying the polymeric network of the material [26]. In addition, leaching of filler particles into oral environment may compromise the biocompatibility of the restorative materials [27].

In this study, composite specimens were first incubated at the constant temperature of 37°C in the various dietary solvents for a 7-days period, prior to undergoing flexural test. This is because in oral environment, there are many unfavourable situations (intermittent exposure: during eating and drinking; continuous exposure: trapping of chemical agents at margins of inappropriately finished restoration and calculus along with food debris that stick to the teeth) in which RBCs are exposed to chemical agents from food and beverages for a longer period of time [10,11,14]. Besides, it had been reported that the hardness of composites which had been conditioned in dietary solvents reduced significantly in the first seven days [28].

As mentioned earlier, flexural strength is an important parameter to signify fracture resistance or the brittleness of restorative materials, and is dependent on the specimens' configuration and ability to withstand load [29]. A higher flexural strength indicates that a material has higher fracture resistance. In other words, higher stress could be withstood by the material before it fails [7]. This mechanical property is clinically relevant as it measures the materials' behaviour in high-stress bearing area, especially in posterior teeth [29].

In dental practice, different flexural properties are required depending on different clinical situations. Restorative materials with low flexural modulus are preferred for Class V cavities as they can flex with the teeth upon occlusal loading, thus preventing displacement during chewing action. On the flip side, the utilisation of materials with relatively higher flexural modulus and strength is required in occlusal and proximal cavities to withstand occlusal forces during mastication and therefore, to prevent fracture [29,30].

The ISO4049 standard specifies that the flexural strength of all dental polymer-based restorative materials have to be at least 80MPa [31]. Except for

HN and AE which had been conditioned in ethanol and HN in artificial saliva, all RBCs demonstrated higher flexural strength than the ISO4049 standard irrespective of the media. Among various RBCs tested, only SF2 demonstrated flexural strength higher than the ISO4049 standards of 80MPa for all media (Air: 106.60MPa; Artificial saliva: 84.45MPa; Citric acid: 88.10MPa; Ethanol: 87.35MPa). This result was consistent with another study in which SF2 showed favourable outcomes in creep recovery behaviour even after immersion in FSLs [26]. The highest flexural modulus was also reported for SF2 in various dietary solvents. This could be due to the higher filler content of SF2 (83.5% Wt) compared to HN and AE (81% Wt). This was validated with studies which showed that higher modulus was associated with higher filler content [11,32]. Lower water absorption was also observed with higher filler loading [33], which has positive effects on structural stability and mechanical properties [34]. Besides higher filler loading, the addition of quartz filler (silicone dioxide) in SF2 caused the material to be less susceptible to aqueous attack, thus having higher values in all storage media [10].

According to the manufacturer, SF2 is a sonic activated single-step high viscosity bulk filled composite and claims up to a 5mm depth of cure in posterior restorations. It is a highly filled resin with special modifiers that reduce its viscosity on sonic activation to allow quick placement and precise adaptation to cavity walls and returns to a more viscous, non-slumping state when sonic activation is stopped. It allows more efficient placement of the restoration, particularly in posterior cavities, thus reducing the procedural time. This is useful in restorative treatment for anxious patients and children where procedure duration should be ideally kept short [35]. Furthermore, SF2 has proven to demonstrate greater flexural strength values, good adaptation to cavity wall, lower polymerisation shrinkage and less microleakage aside from creation of less voids due to the injection technique used [16,35,36].

Nevertheless, AE recorded the greatest value of flexural strength after storing in air, artificial saliva and citric acid. This could be correlated with the use of diurethane dimethacrylate (UDMA) in combination with TEGDMA in the formulations of AE which has positive effects on its mechanical properties [37]. The addition of UDMA as comonomer in dental RBCs produces polymers with low polymerisation shrinkage, increased hydrophobicity, decreased water degradation and good mechanical properties [38]. However, its flexural properties were significantly affected by

ethanol. This could be due to lower filler loading in AE, despite the addition of more hydrophobic UDMA, that does not render it resistant to water degradation when exposed to extreme dietary condition simulated by ethanol. This could be explained by variation in RBCs' chemical composition and their filler characteristics such as particles size, shape and distribution lead to variation in their properties [10].

All materials conditioned in air demonstrated the highest flexural strength among various media. This result was consistent with several studies [11,30]. This can be explained by non-leakage of silica and filler particles with conditioning in air in contrast to storage in aqueous media [15]. Other than SF2, tested RBCs which were immersed in ethanol presented the smallest value of flexural strength. Weakening of RBCs is due to the fact that ethanol promotes plasticisation of the polymeric matrix by dissolving and pulling apart the residual monomers and linear polymers in the polymer structure [21]. Therefore, it may be suggested that alcohol containing drinks may decrease the durability of the restoration. Thus, it may be prudent to take dietary habits of patients into considerations in order to choose the right material wisely and to improve the functional longevity of restoration. Besides, this clinical finding could support the need for dietary advice to be routinely given to patients after restoration.

Our studies had limitations. First of all, the constant conditioning temperature at 37°C and the static test does not simulate temperature fluctuation in the oral cavity. Temperature in the oral cavity experienced during routine eating and drinking ranges from 0°C to 70°C [39]. These temperature fluctuations will affect the clinical performance of restorative materials in the oral environment [9]. Another limitation is the nature of testing used. Static three-point bending may lead to undesirable edge failure of specimen evaluated, causing misleading data [3]. Static flexural testing performed in this study gives limited information on the material structure due to the visco-elastic nature of dental composites. The use of static testing has its limitations compared to dynamic testing. Dynamic testing with dynamic mechanical analysis (DMA) could be conducted as it is useful for accessing both viscous and elastic characteristics of

dental composites, thus being more clinically relevant than the static one [8,40]. As restorative materials are subjected to dynamic loading and fluctuation of temperature intraorally, the implementation of variation of temperatures, frequencies and displacement in DMA can well reflect the clinical situation in the oral environment. Furthermore, the non-destructive nature of dynamic testing allows the specimens to be re-examined over a longer period of time [40]. In addition, since only flexural properties were evaluated in this study, the evaluation of other physico-mechanical, biological and chemical properties such as hardness, wear, colour stability and fluoride release should be carried out in future studies to fully characterise the clinical performances of resin-based composites under dietary challenges. Besides, the results obtained from these laboratory studies have to correspond to results of clinical trials [41].

CONCLUSION

The impact of dietary solvents on flexural properties were dependent on materials and conditioning media. Immersing in air (control) usually presented the highest flexural properties. Conditioning in aqueous solution (artificial saliva, citric acid and ethanol) generally contributed to reduced flexural properties. Other than SF2's flexural modulus, ethanol significantly decreased the RBCs' flexural properties. These findings provide support for clinicians to advice their patients to reduce the consumption of certain food and beverages for the clinical success of dental restoration. Also, clinicians have to consider the patients' dietary habits in order to choose the right material wisely for long term functional longevity of restoration.

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DECLARATION OF INTEREST

The authors report no conflicts of interest in this work.

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