

Assessment the effect of acid and base cycling on mechanical properties of various esthetic restorative materials

Abstract

Objective: The purpose of this study is the evaluation of the effect of pH cycling, including both acidic and alkaline environments, on the mechanical properties of tooth-colored restorative materials.

Methods & Materials: 20 rectangular bar specimens of one bulk fill restorative composite; (Tetric N Ceram bulk fill), two conventional universal nanohybrid restorative composites; (Grandio, and Clear fill APX ES2), and one restorative resin-modified glass ionomer; (Fuji II LC) were produced according to ISO 4049. Half of the materials were stored in a pH cycling regimen defined as two-day storage in acidic (pH =4) and alkaline (pH=8) solutions. The rest of the materials were incubated in distilled water as a control group. The storage lasted for 48 days. Finally, flexural strength, elastic modulus, and microhardness of the specimens in each group determined. Data analyzed with Kruskal-Wallis, Dunn, MANOVA, Tukey HSD and T-test.

Results: The pH cycling model had a significant influence on all mechanical properties of the Tetric N Ceram bulk fill and RMGI than those stored in water. Clear fill APX ES2 showed a significant reduction in elastic modulus and microhardness while Universal Nanohybrid Restorative Grandio showed a significant reduction only in flexural strength.

Conclusion: pH cycling negatively affects the mechanical properties of tooth-colored restorative materials, and the materials' composition is an important factor in the degradation of the resin-based materials examined.

Keywords: resin composite; bulk fill; mechanical properties

Introduction

Tooth-colored restorative materials are primarily used restorative materials in dental practice (1), and improving their clinical performance is a focal point of new research. In this regard, one important aspect is increasing hydrolytic stability. Chemical hydrolysis of tooth-colored restorative materials occurs in water, which hydrolyzes the condensation type bonds (2). Two types of categories influence the hydrolysis rate. The first category includes factors that are related to the environment like acids(3), bases (4), enzymes (5) and etcetera. The second category relates to the structural factors of the tooth-colored materials like the type of the resin matrix(6), filler content(7), filler type(8), and filler size(9).

Since evaluating all these factors' simultaneous effects is difficult in the laboratory, many researchers have to reproduce a restricted form and work on models. Some researchers use a pH cycling regimen that usually contains acidic pH since most of the beverages have pH below 4(10), and regular intake of fermentable carbohydrates leads to acidic pH as low as 3.9 in the dental biofilm(11). There is conflicting evidence whether the effect of hydrogen ion on hydrolysis rate is primarily from the matrix, the filler particles, or the interfacial-coupling agent. According to the study which is done by Soderholm et al., hydrogen ions possibly accelerate ion exchange at the filler surface. Tooth-colored resin restorations, when are stored in water, leach Alkaline-earth elements such as Strontium and Barium. Leaching of these positive ions leaves the remaining Si-O-Si structures negatively charged, and the charge imbalance could delay the release of more ions on the surface. However, positive ions such as H^+ in the environment could help neutralize the negative charges and facilitate further leaching of glass modifiers (12, 13).

Evaluation of the effect of an alkaline environment on tooth-colored restorative materials is not sufficient. However, it has been proposed that leaching of glass modifiers results in OH ion production at the resin-filler interface. Charles proposed that this ion hydrolyzes the

silanol bonds and makes the reaction autocatalytic(13). It is reported that the pH of this alkaline environment for barium-containing glasses at the filler interface is 9.3 (4).

Therefore, OH and H ions theoretically have different action mechanisms on dental resin materials. However, they both lead to the gradual destruction of fillers, affecting dental resin restorations' mechanical properties. However, most studies have not evaluated the effect of different pH mediums on dental resin materials' mechanical properties. Most studies had been confined to surface roughness, microhardness, water sorption, and solubility(4, 14-16). In this way, the present research aims to evaluate the interaction between resin-based restorative materials, including some recent materials and acid and base cycles. The null hypothesis tested were no differences in the flexural strength, elastic modulus, and microhardness of recent tooth-colored restorative materials after storage in different pH media over time.

Methods & Materials

1. preparation of samples

Three resin-composite materials, including one bulk fill and two conventional materials, and one resin-modified glass ionomer are tested. Product specifications are presented in Table 1.

Table 1. Product specifications of materials evaluated

Materials	Abbreviation	Manufacturer	Shade	Filler Weight (%)	Filler type	Filler size (μ)	Matrix
Tetric N- ceram bulk fill(nanohybrid)	TNB	Ivoclarvivadent	IVA	75-77	barium glass, prepolymer filler	0.04to 3	Bis-GMA, UDMA
Universal nanohybridGrandio	GR	Voco	A2	87	silica glass	0.02 to 1	TEGDMA, Bis-GMA
Clearfil APX ES-2 (universal, nanohybrid)	CL	Kuraray	A2	86	Silanated barium glass, Silanated silica, Silanated colloidal silica	0.02 to 17	TEGDMA, Bis-GMA
GC Fuji II LC	RMGI	GC America	A2	Distilled water, polyacrylic acid,			

(Resin modified glass ionomer cement)				2-hydroxyethylmetacrylate, urethane dimethacrylate, camphorquinone, fluoroaluminosilicate filler
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A rigid transparent acrylic bar was prepared in 2×2×25 mm dimensions according to ISO 4049:2019 by water cooling three-axis CNC milling machine. A mold was then made by pressing the plastic specimen into the silicone impression putty (Speedex, Coltene).

Twenty rectangular bar specimens of each tooth-colored restorative material were prepared at room temperature (23±2°C). The mold was slightly overfilled with uncured material in a single increment. A polyester strip with a glass slab was placed over the mold to obtain a flat surface and standardize the curing distance by placing the light tip in contact with the glass. As the sample length was 25 mm, and the light exit diameter was 5 mm, each sample was polymerized in five overlapping sections for 20 seconds per section in a pulse curing mode. The calibrated light-curing unit (CL37, Jerry, China) had an irradiance of 1000 mW/cm², and wavelength ranges were about 420–480 nm. After curing, each sample was gently removed from the mold, and specimens with any cracks or voids were replaced. The excess minor materials were removed carefully by a sharp blade at the periphery, and no polishing was performed. Final specimens were pre-stored in distilled water at room temperature for seven days.

2. pH-storage regimen

The specimens were then randomly divided into experimental and control groups (n= 10). The experimental group was stored in a pH cycling, and the control group was immersed in distilled water. The pH cycling was defined as two-day storage in acidic solution (pH=4) and two-day storage in alkaline solution (pH=8). After every two days, solutions were refreshed, and samples in the experimental group were rinsed with distilled water before entering the next solution. The pH solutions were prepared based on the citrate-phosphate buffer

(McIlvanes standard buffer solution) described by Ortengren et al. (15). Samples were kept in sealed glass containers in an incubator at 37 °C for 48 days.

3. mechanical properties evaluations

After storage, a universal testing machine (SANTAM STM-20) in three-point bending mode was used to measure the flexural strength (FS - MPa) and flexural modulus (FM - GPa) of all samples until fracture occurred. The crosshead speed was 1 mm/min, and the test span was 15 mm. Flexural strength (σ) and elastic modulus (E) were calculated as follows:

$$\sigma = 3PL/2bh^2$$

Where P is the peak load from the load–displacement curve in N, L is the span in mm, b is the specimen width in mm, and h is the specimen height in mm.

$$E = PL^3/4bh^3d$$

Where P is the maximum load in N, L is the span in mm, b is the specimen width in mm, h is the specimen height in mm, and d is the deflection corresponding to the load P in mm.

Microhardness measurements were performed on the samples' top surface by a diamond pyramid indenter using a Vickers microhardness tester (MH3, Koopa, Iran). It was applied 0.98N load for 10s on the upper surface, and the average value of the two indentation diagonals was measured using a microscope. Two measurements were taken and averaged. All measurements were carried out at a constant room temperature.

4. statistical analysis

Statistical analyses were performed using SPSS Statistics 21.0 and Microsoft Excel 2016. Assuming four types of materials and two storage mediums (distilled water and pH cycling),

mechanical properties data were analyzed for eight groups. The normality of data distribution was verified by Shapiro-Wilk. Experimental groups' data were not homoscedastic. Therefore, Kruskal-Wallis was applied to determine the mean values of mechanical properties, and Dunn's test was applied for pairwise comparison of materials' mechanical data. Control groups' data were homoscedastic. Therefore, MANOVA was used to determine the mean values of mechanical properties, and Tukey HSD was used for pairwise comparison of materials' mechanical data. T-test was used to identify significant inter-solution differences in mechanical properties for each restorative material. In all analyses, the level of significance was justified at $p < 0.05$.

Results:

Table 2 shows the mean flexural strength, flexural modulus, and microhardness of each group after 48 days. Grandio showed a significant reduction in flexural strength, while Clear fill showed a significant reduction in elastic modulus and microhardness. All mechanical properties of Tetric N Ceram bulk fill and RMGI reduced significantly than those stored in distilled water. Most of the materials' mechanical data in distilled water were still significantly different after pairwise comparison (table 3). However, pairwise comparison of the materials' mechanical data after the pH cycling regimen shows statistical differences between GR-TEC, GR-RMGI, and CL-RMGI (table 4).

Table 2. Mean values for flexural strength, flexural modulus, and microhardness in experimental and control groups

groups	flexural strength (MPa)	flexural modulus (GPa)	microhardness(VH)
	Mean(\pm SD)		
TNB	51.72(15.54) ^C	31.77(5.41) ^C	2041.12(1315.50) ^C
TNB (control)	94.73(11.12) ^c	50.89(3.61) ^c	4042.94(1212.61) ^c
GR	114.34(8.67) ^A	111.26(8.42) ^A	6522.71(1124.39) ^A
GR (control)	132.79(18.34) ^a	107.64(8.36) ^A	6407.75(1225.67) ^A
CL	86.31(17.52) ^B	52.8(4.11) ^B	2831.38(628.43) ^B
CL (control)	87.34(19.63) ^B	79.73(3.16) ^b	4175.02(757.88) ^b

RMGI	13.5(13.34) ^D	14.07(2.85) ^D	1151.71(328.23) ^D
RMGI (control)	56.99(14.31) ^d	33.48(2.81) ^d	2766.68(660.93) ^d

Same superscript upper case letters indicate no statistical significance among values for each tooth-colored restorative material. SD, standard deviation. GR, Grandio. CL, Clearfill. TNB, Tetric N cerambulkfill. RMGI, Resin modified glass ionomer.

Table 3. pairwise comparison of flexural strength, elastic modulus and, microhardness of restorative materials in distilled water (control group)

materials	Mean difference	p-value	95% confidence interval	
			Lower Bound	Upper bound
Flexural strength (MPa)				
GR-CL	45.44	0.000	25.92	64.96
GR-TEB	38.05	0.000	18.53	57.57
GR-RMGI	75.80	0.000	56.28	95.32
CL-TEB	7.38	0.739	26.91	12.13
CL-RMGI	30.35	0.001	10.83	49.87
TEB-RMGI	37.74	0.000	57.26	18.22
(GPa)Elastic modulus				
GR-CL	27.91	0.000	33.96	21.86
GR-TEB	56.75	0.000	62.80	50.70
GR-RMGI	74.16	0.000	80.21	68.11
CL-TEB	28.84	0.000	34.89	22.79
CL-RMGI	46.25	0.000	52.30	40.20
TEB-RMGI	17.41	0.000	11.36	23.46
Microhardness(VH)				
GR-CL	2232.72	0.000	3434.75	1030.69
GR-TEB	2364.80	0.000	3566.83	1162.77
GR-RMGI	3641.06	0.000	4843.08	2439.03
CL-TEB	132.08	0.991	1334.10	1069.94
CL-RMGI	1408.33	0.016	2610.36	206.30
TEB-RMGI	1276.25	0.034	74.22	2478.28

GR, Grandio. CL, Clearfill. TNB, Tetric N cerambulkfill. RMGI, Resin modified glass ionomer.

Table 4. pairwise comparison of flexural strength, elastic modulus and, microhardness of restorative materials in pH cycling solutions (experimental group)

Materials	Mean(±SD)		p-value
Flexural strength (MPa)			
GR-CL	8.80	1.68	0.554
GR-TEB	18.50	3.53	0.002
GR-RMGI	29.10	5.56	0.000
CL-TEB	9.70	1.85	0.381
CL-RMGI	20.30	3.88	0.001
TEB-RMGI	10.60	2.02	0.256
(GPa)Elastic modulus			
GR-CL	10.00	1.91	0.335
GR-TEB	20.00	3.82	0.001
GR-RMGI	30.00	5.73	0.000
CL-TEB	10.00	1.91	0.335
CL-RMGI	20.00	3.82	0.001
TEB-RMGI	10.00	1.91	0.335
Microhardness(VH)			
GR-CL	12.70	2.42	0.091

GR-TEB	19.80	3.78	0.001
GR-RMGI	27.10	5.18	0.000
CL-TEB	7.10	1.35	1.00
CL-RMGI	14.40	2.75	0.035
TEB-RMGI	7.30	1.39	0.976

GR,Grandio. CL,Clearfill. TNB,Tetric N cerambulkfill. RMGI,Resin modified glass ionomer.

Discussion:

As shown by the present findings, all mechanical properties of TNB were more susceptible to pH cycling than conventional composites. TNB has lower filler content and contains barium glass and pre-polymerized fillers. Pre-polymerized fillers may facilitate water and ion transportation inside dental resin materials. Water sorption results in resin matrix swelling, but the swelling of the pre-polymerized fillers is retained. Since the bonding between the pre-polymerized fillers and the matrix may not be optimized, gaps will open, acting as pathways and letting the interaction between internal fillers and the surrounding medium (17). There is also evidence that water sorption of dental resin restorations is mainly associated with the polymeric matrix, and tooth-colored resin materials with lower filler content absorb more water than the opposite (6). It was reported that higher filler content could improve composites' resistance against acidic mediums. They evaluated the effect of composites' filler content on composites' resistance to morphological changes in acidic mediums. After 14 days, composites' morphological changes were observed by SEM and confocal laser scanning microscope, and composites with higher filler loadings generally seemed to have a higher resistance to acidic mediums. However, a linear relationship could not be observed and, filler types, filler distribution, and surface treatment of fillers were also important factors (7).

Another important consideration is that the TNB resin matrix is more hydrophobic than GR and CL resin matrixes. Alshali et al. previously conducted a comparison between all these factors. They compared water sorption of TetricEvo Ceram bulk fill and Grandio SO in distilled water for up to a year. Finally, water sorption of both composites related to their resin matrix was not statistically significant (GrandioSO=4.05% SPpm, TetricEvo Ceram

Bulk Fill=4.26% SPpm). However, the general water sorption of TetricEvo Ceram bulk fill was statistically greater than Grandio SO water sorption, which could be attributed to its lower filler content(6). Their results concur with those of Alrahlah et al. who conducted an almost identical study(18). Therefore, based on the present findings, it may be deduced that the higher susceptibility of TNB than other evaluated composites is due to the lower filler weight and incorporation of pre-polymerized fillers and glass modifiers.

The flexural strength of GR and elastic modulus and microhardness of CL reduced significantly in the pH-cycling model than those of the distilled water. Therefore, it can be proposed that CL had been more susceptible to pH challenge than GR. CL and GR have nearly identical structures except in the filler type, in which GR contains silica glasses purely, while CL contains silica glasses plus Barium glass modifiers. There is evidence that glass modifiers often reduce filler particles' hydrolytic stability(8, 13). They accelerate siloxane bond hydrolysis. Alkaline-earth elements incorporated in fillers leach from the filler particles, react with water, and make the surroundings alkaline. When a particular hydroxyl ion concentration is reached, these hydroxyl ions could start attacking the silica network and make the siloxane bond hydrolysis automatic(13).

However, more studies showed that the storage medium's effect is more pronounced than the effect of filler type (19). Continuity of the explained mechanism is dependent on the ion exchange with the environment. Leaching of Alkaline-earth elements forms negative charges within the silicone network. Such negative charges could delay further leaching of these ions retained inside the network until a charge balance is re-established. Such a charge balance could occur if positive ions diffuse from the environment to the filler surface and neutralize the negative charges. Thus by neutralizing the filler particle charges, Alkaline-earth elements otherwise retained within the filler particles would be more readily available for further release (12).

The present study used pH cycling, containing both acidic and alkaline environments. Based on the above explanations, it can be assumed that hydrogen ions in an acidic medium

accelerate the leaching of barium from CL since they penetrate the spaces previously occupied by barium and maintain the charge balance inside the silica. However, theoretically, it seems that in an acidic medium, the autolytic degradation of CL and GR glass fillers by hydroxyl ions stops. Contrariwise, in an alkaline medium, the charge imbalance restricts the leaching of barium from CL. A million times as many hydroxyl ions as would be produced from barium glasses are present in the solution, and the autocatalysis of CL and GR siloxane bonds by hydroxyl ions intensifies. Therefore, surface fillers of tooth-colored resin materials become improperly bonded gradually, and as a result of such a debonding, the mechanical properties will decrease substantially.

Tarumi et al. evaluated the effect of filler type and filler size on the flexural strength of composites after one-week water immersion. Finally, they showed the flexural strength of barium-containing resin decreased after water immersion more than that of the quartz-containing resin as the particle size decreased. Several studies report similar findings regarding the lowering effect of glass modifiers on resin-based restorative materials' different physical properties ([4](#), [15](#), [20](#)).

Resin modified glass ionomer had the lowest mechanical data in both experimental and control groups than composite resins. Their mechanical properties reduced significantly in the pH-cycling regimen than in distilled water. These findings could be explained by differences in the physical chemistry of resin-modified glass ionomers and resin composites.

Resin-modified glass ionomer consist of aluminum fluorosilicate glasses and modified polyacrylate networks cross-linked by poly-HEMA. It is proposed that the coherence between the polyacrylate acid network and the poly-HEMA may be insufficient, and gaps will form as a result of phase separation([21](#)). Besides, the poly-HEMA is a water-based resin system that contains a high proportion of hydrophilic functional groups and takes up a lot of water([22](#)). Therefore, water and H⁺ and OH⁻ ions can more easily diffuse into the matrix and provoke ionic species' dissolution from the glass particles. Consequently, there is a gradual reduction in the mechanical properties of RMGI. Moreau et al.compared flexural strength and elastic

modulus changes of Fuji II LC and Heliomolar in lactic (pH=4) or acetic (pH=5.5) acids after 84 days. Results showed that immersion decreased mechanical properties, significantly for Fuji II LC compared to Heliomolar' (23).

Changes in the RMGI microhardness coincide with other studies evaluating RMGI microhardness changes in acidic mediums (24-26). However, Gomec et al. evaluated surface microhardness of different tooth-colored restorative materials in different acidic solutions for a week and showed contrary results. The microhardness of composite reduced in all acidic media; however, the microhardness of RMGI increased in orthophosphoric acid. Authors have attributed the higher hardness values to the deposition of insoluble salts like calcium phosphate and aluminum phosphate with high ionic bonding and low solubility on the surface of RMGI (27). However, in the present study, citrate-phosphate buffer solution was used, and citric acid has large complexing ability with metal actions that may inhibit insoluble salts' deposition on RMGI. Nicholson et al. evaluated gain in the mass of different tooth-colored restorative materials in different acidic solutions and showed that citric acid was an especially aggressive medium towards these materials, particularly for glass-ionomer cement (28).

Regarding the effect of alkaline medium on glass ionomers, two studies were evaluated, and the results suggested that glass ionomers are more prone to alkaline medium than acidic medium. Yanikoğlu et al. studied the solubility of different dental cement in different pH mediums for a month. The solubility of glass polyalkenoate ionomer (Voco, Germany) in alkaline medium (pH=9) was higher than that of acidic medium (pH=3) (29). Bagheri et al. evaluated surface degradation of composites and compomers stored in different pH mediums by using a silver nitrate staining technique and taking SEM photographs to measure the depth of silver penetration. Samples were incubated in lactic acid (pH=4) or NaOH (pH=13) or distilled water for two weeks. Results showed the degradation of all materials in all chemical media, but to a significantly greater depth of degradation after exposure to NaOH media. However, composites were not necessarily more resistant than compomers, which shows the importance of the materials' composition (20).

According to the mechanical properties tested, the flexural testing was conducted in three-point loading due to the more comfortable application. However, it has been shown that flexural strength values are statistically higher in three-point bending than in four-point bending (30). Besides, the span length in the universal testing machine was considered 15 mm. However, according to ISO 4049 flexural test (IFT) for samples in 2×2×25 mm dimensions, the span length should be 18.8 mm (31). Also, in the Vickers microhardness tester, the optical reading of the diagonals was not automatic and was subject to the problems of operator influence.

Limitations of this study were non-inclusion of other essential factors on the chemical hydrolysis of resin-based restorative materials to create a more clinically relevant testing environment. It must be mentioned that the results from this study were generated under conditions that were far from clinical practice. Besides, separated acidic and alkaline mediums should have been used, and the number of samples should have been increased. Therefore, future research should consider the evaluation of newly introduced restorative materials since the effect of different pH mediums in long term on the tooth-colored restorative materials seems to be material dependent. Besides, to proceed further in improving material performance, more investigations should focus on the effect of type of filler, resin matrix, and pre-polymerized fillers and coupling agents on the materials' mechanical properties in different pH media accompany other aging factors.

Conclusion:

In light of the presented results, it can be concluded that pH cycling negatively affects the mechanical properties of tooth-colored restorative materials. However, the extent of the effect depends on the materials' composition. Filler loading and incorporation of glass modifiers and pre-polymerized fillers are some structural factors of materials that could have been important.

COMPETING INTERESTS DISCLAIMER:

Authors have declared that no competing interests exist. The products used for this research are commonly and predominantly use products in our area of research and country. There is absolutely no conflict of interest between the authors and producers of the products because we do not intend to use these products as an avenue for any litigation but for the advancement of knowledge. Also, the research was not funded by the producing company rather it was funded by personal efforts of the authors.

References

1. Moosavi H, Zeynali M, Pour ZH. Fracture resistance of premolars restored by various types and placement techniques of resin composites. *Int J Dent* 2012.
2. Pinna R, Usai P, Filigheddu E, Garcia-Godoy F, Milia E. The role of adhesive materials and oral biofilm in the failure of adhesive resin restorations. *Am J Dent*. 2017;30:285-92.
3. Scribante A, Bollardi M, Chiesa M, Poggio C, Colombo M. Flexural properties and elastic modulus of different esthetic restorative materials: evaluation after exposure to acidic drink. *BioMed Res Inte*. 2019;2019.
4. Cilli R, Pereira JC, Prakki A. Properties of dental resins submitted to pH catalysed hydrolysis. *J Dent*. 2012;40:1144-50.
5. Bourbia M, Ma D, Cvitkovitch DG, Santerre JP, Finer Y. Cariogenic bacteria degrade dental resin composites and adhesives. *J Dent Res*. 2013;92:989-94.
6. Alshali RZ, Salim NA, Satterthwaite JD, Silikas N. Long-term sorption and solubility of bulk-fill and conventional resin-composites in water and artificial saliva. *J Dent*. 2015;43:1511-8.
7. Han L, Okamoto A, Fukushima M, Okiji T. Evaluation of flowable resin composite surfaces eroded by acidic and alcoholic drinks. *Dent Mater J*. 2008;27:455-65.

8. TARUMI H, TORII M, TSUCHITANI Y. Relationship between particle size of barium glass filler and water sorption of light-cured composite resin. *Dent Mater J*. 1995;14:37-44,102.
9. Karabela MM, Sideridou ID. Synthesis and study of properties of dental resin composites with different nanosilica particles size. *Dent Mater*. 2011;27:825-35.
10. Reddy A, Norris DF, Momeni SS, Waldo B, Ruby JD. The pH of beverages available to the American consumer. *J Am Dent Assoc*. 2016;147:255.
11. Bowen WH. The Stephan curve revisited. *Odont*. 2013;101:2-8.
12. Söderholm KJM, Yang MC, Garcea I. Filler particle leachability of experimental dental composites. *Eur J Oral Sci*. 2000;108:555-60.
13. Soderholm K-J, Mukherjee R, Longmate J. Filler leachability of composites stored in distilled water or artificial saliva. *J Dent Res*. 1996;75:1692-9.
14. Chadwick R, McCabe J, Walls A, Storer R. The effect of storage media upon the surface microhardness and abrasion resistance of three composites. *Dent Mater*. 1990;6:123-8.
15. Ortengren U, Andersson F, Elgh U, Terselius B, Karlsson S. Influence of pH and storage time on the sorption and solubility behaviour of three composite resin materials. *J Dent*. 2001;29:35-41.
16. Moosavi H, Ghavamnasiri M, Manari M. Effect of postoperative bleaching on marginal leakage of resin composite and resin-modified glass ionomer restorations at different delayed periods of exposure to carbamide Peroxide. *J Contemp Dent Pract*. 2009, 10, E009-16.
17. Soderholm K-J. Leaking of fillers in dental composites. *J Dent Res*. 1983;62:126-30.
18. Alrahlah A. Physical, Mechanical and Surface Properties of Dental Resin-composites: The University of Manchester (United Kingdom); 2013.

19. Drummond JL, Andronova K, Al-Turki LI, Slaughter LD. Leaching and mechanical properties characterization of dental composites. *Journal of Biomedical Materials Research Part B: Applied Biomaterials: An Official Journal of The Society for Biomaterials, The Japanese Society for Biomaterials, and The Australian Society for Biomaterials and the Korean Society for Biomaterials.* 2004;71:172-80.
20. Bagheri R, Tyas MJ, Burrow MF. Subsurface degradation of resin-based composites. *Dent Mater.* 2007;23:944-51.
21. Turssi C, Hara A, Serra M, Rodrigues Jr A. Effect of storage media upon the surface micromorphology of resin-based restorative materials. *J Oral Rehabil.* 2002;29:864-71.
22. Kanchanasavita W, Anstice H, Pearson GJ. Water sorption characteristics of resin-modified glass-ionomer cements. *Biomater.* 1997;18:343-9.
23. Moreau JL, Xu HH. Fluoride releasing restorative materials: Effects of pH on mechanical properties and ion release. *Dent Mater.* 2010;26:e227-e35.
24. Hengtrakool C, Kukiattrakoon B, Kedjarune-Leggat U. Effect of naturally acidic agents on microhardness and surface micromorphology of restorative materials. *Eur J Dent.* 2011;5:89.
25. Hamouda IM. Effects of various beverages on hardness, roughness, and solubility of esthetic restorative materials. *J Esth Rest Dent.* 2011;23:315-22.
26. Aliping-McKenzie M, Linden R, Nicholson J. The effect of Coca-Cola and fruit juices on the surface hardness of glass-ionomers and 'compomers'. *J Oral Rehabil.* 2004;31:1046-52.
27. GÖMEÇ Y, DORTER C, ERSEV H, EFES BG, YILDIZ E. Effects of dietary acids on surface microhardness of various tooth-colored restoratives. *Dent Mater J.* 2004;23:429-35.
28. Nicholson J, Gjorgievska E, Bajraktarova B, McKenzie M. Changes in properties of polyacid-modified composite resins (compomers) following storage in acidic solutions. *J oral Rehabil.* 2003;30:601-7.

29. Yanikoglu N, Duymus ZY. Evaluation of the solubility of dental cements in artificial saliva of different pH values. *Dent Mater J.* 2007;26:62-7.
30. Calabrese L, Fabiano F, Bonaccorsi LM, Fabiano V, Borsellino C. Evaluation of the clinical impact of ISO 4049 in comparison with miniflexural test on mechanical performances of resin based composite. *IntJ Biomater.* 2015;2015.
31. Chitchumnong P, Brooks S, Stafford G. Comparison of three-and four-point flexural strength testing of denture-base polymers. *DentMater.* 1989;5:2-5.

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