

# Effects of Storage Media on Physical Properties of Selected Tooth Coloured Restorative Materials

Leili Sadaghiani\*, Gabriel Adusei and Jeremy Rees†

**Abstract** - It is known that storage media can affect the physical properties of some restorative dental materials. The purpose of this laboratory study was to investigate the possible effects of storage media on physical properties of a conventional glass-ionomer, a resin modified glass ionomer and a compomer. Specimens of the restorative materials in the study (FujiII LC, FujiIX and Dyract EXTRA) were prepared. The specimens were stored in either water or artificial saliva with or without exposure to Listerine. The compressive and diametral tensile strength and Vickers hardness of these materials were tested at 24 hours, 1 week, 4 weeks and 12 weeks. Compressive and diametral tensile strength for FujiII LC and Fuji IX had increased at 12 weeks. A decrease was observed for Dyract EXTRA in the same period. No significant differences were observed between the storage media ( $P>0.01$ ). Vickers hardness values fluctuated during the testing period, with a pattern being consistent for each material. Storage of materials investigated for the period in this study resulted in superior compressive and diametral tensile strength for Fuji II LC and FujiIX. The opposite was true for Dyract EXTRA. Effects of time were found to be more pronounced than the media ( $P<0.01$ ).

KEY WORDS: Storage media; Tooth-coloured restorative materials; Compressive strength; diametral tensile strength, Vickers hardness

## INTRODUCTION

The oral environment is a hostile place and presents restorative dental materials with factors that may affect their physical properties and clinical performance. Interactions with saliva and other aqueous-based liquids such as mouthwashes, alcoholic and acidic drinks are examples of such factors. Polymeric and poly-acid based restorative materials undergo changes in different storage media which may include volumetric and dimensional transformations affecting properties such as strength, flexural modulus and hardness<sup>1-5</sup>. In general, the mechanical properties of restorative materials such as glass-ionomer cements increase with water storage, whereas the strength of resin-based materials may decrease when stored in water<sup>6</sup>.

Regular use of a mouthwash in combination with tooth brushing and flossing have been recommended by many clinical studies as effective oral hygiene practice and help in reducing plaque build up and periodontal diseases<sup>7,8</sup>. It is necessary for the dental profession to understand the possible effects of mouthwashes on the oral hard and soft tissues and the major groups of restorative materials prior to giving advice to patients.

Amongst the various studies conducted on the effects of storage media on the properties of restorative dental materials, very few have considered the effect of intermittent exposure of materials to mouth rinses. A recent study revealed that exposure of resin modified glass ionomers to certain mouthwashes had adverse effects on surface roughness<sup>9</sup>.

The aim of this study was to evaluate the effect of different storage media on selected physical properties of three commercial dental restorative materials.

## MATERIALS AND METHODS

Three commercial dental restorative materials were examined; Fuji II LC, Fuji IX and Dyract EXTRA (Table1). The tests employed to evaluate mechanical properties were compressive strength, diametral tensile strength and Vickers hardness. Cylindrical specimens of 4mm diameter and 6mm in height [ISO 9917-1:2003] were prepared in stainless steel moulds and used for Vicker hardness, diametral tensile and compressive strength tests. Wax (Chesebrough-Ponds Ltd., London, UK) was applied to the moulds for lubrication. For FujiII LC and FujiIX, specimens were mixed according to the liquid-powder ratio recommended by the manufacturer. Moulds were filled with paste in excess and pressed with two glass slides at each end and excess paste was removed. The specimens of Fuji II LC and Dyract EXTRA were then exposed to a curing light, using a blue halogen light-curing unit, for 20 seconds with Prismetic Lite II (Dentsply DeTrey, Konstanz, Germany) from each end of specimen. For Fuji IX, moulds were filled with the mixed cement paste and then covered with clear cellulose matrix strips for 1 hour, kept at room temperature, before being placed in the storage media.

One hundred and sixty eight specimens for each material were prepared and divided into groups of seven to be placed in one of the three storage media for testing at 24hrs, 1 week, 4 weeks and 12 weeks. The storage media used were double distilled water or artificial saliva with or without intermittent exposure to Listerine (Pfizer Consumer Healthcare, Surrey, UK) mouthwash. The active ingredients, alcohol content and pH of Listerine are given in Table 1.

\* DDS, MSc

† BDS, MScD, PhD

**Table 1.** Restorative materials and mouthwash used in the study

Material		Manufacturer	
Fuji II LC		GC Corporation, Tokyo, Japan	
Fuji IX		GC Corporation, Tokyo, Japan	
Dyract EXTRA		DENTSPLY DeTrey GmbH, 78467 Konstanz, G, Germany	
Mouthwash	Active ingredients	pH	Alcohol %
Listerine	Ethanol, Benzoic acid, Eucalyptol, Menthol, Methylsalicylate, Thymol	4.05	29.2

**Table 2.** Composition of the artificial saliva and pH [10]

Constituent	Amount
Sodium Chloride	0.400 gm L <sup>-1</sup>
Potassium chloride	0.400 gm L <sup>-1</sup>
Calcium chloride. H <sub>2</sub> O	0.795 gm L <sup>-1</sup>
Sodium dihydrogen phosphate. H <sub>2</sub> O	0.690 gm L <sup>-1</sup>
Sodium sulphide.9H <sub>2</sub> O	0.005 gm L <sup>-1</sup>
Distilled water	1000ml
pH	5.52

**Table 3.** Compressive strength of materials tested.

Material/ Media	24hr	1w	4w	12w
<i>FujiII LC</i>				
Water	136.98 (5.38)	140.93 (3.93)	144.85 (3.60)	142.83 (6.87)
Saliva	137.26 (6.40)	139.06 (3.96)	136.26 (3.87)	143.56 (5.96)
SL	137.20 (5.48)	139.97 (1.94)	139.20 (4.25)	139.67 (7.23)
Average	137.15 (5.48)	139.94 (3.29)	140.10 (5.21)	142.02 (6.60)
<i>FujiIX</i>				
Water	147.90 a (6.25)	156.40 ab (5.63)	156.13 ab (7.28)	165.89 b (6.60)
Saliva	147.47 a (4.24)	154.23 ab (4.65)	158.60 ab (6.30)	164.83 b (8.52)
SL	149.04 a (6.35)	150.03 a (3.37)	157.73 ab (7.49)	163.44 b (5.95)
Average	148.14 a (5.45)	153.56 ab (5.18)	157.49 b (6.76)	164.72 c (6.82)
<i>Dyract EXTRA</i>				
Water	222.21 a (8.01)	205.21 b (9.08)	186.21 c (5.63)	186.71 c (5.18)
Saliva	211.4 a (9.51)	192.93 b (5.53)	187.91 b (4.85)	183.31 b (5.02)
SL	212.31 a (8.14)	192.49 b (3.51)	185.40 bc (4.25)	176.24 c (6.06)
Average	215.31 a (9.56)	196.88 b (8.61)	186.51 c (4.81)	182.09 c (6.83)

No statistically significant differences between values for FujiII LC ( $P>0.01$ ). For Fuji IX and Dyract EXTRA values sharing the same lower case letters were not statistically different for comparisons within each row ( $P>0.01$ ). No statistically significant differences when comparing values within a column for each material ( $P>0.01$ ).

The exposure to mouthwash was that recommended by the manufacturer, twice daily for 1 minute.

The artificial saliva used was based on formulation described by Fusayama et al <sup>10</sup> (Table 2). For each material, all sets of specimens were stored in their respective media in a sealed plastic container at 37°C.

The compressive strength (CS) and diametral tensile strength (DTS) were conducted using a Lloyd LR-10K (Lloyd Instruments Ltd, Fareham, Hants, UK) universal testing machine fitted with a 1 kN load cell, operating at a cross-head speed of 1 mm min<sup>-1</sup>. Hardness values were recorded in Vickers Hardness (VHN) kg/mm<sup>2</sup>. The measurements were taken using a Vickers hardness tester (MVK-G1, Mitutoyo Corporation, Tokyo, Japan), using a 500 gram load and a 5 second dwell time. For each specimen, 2 indentations were made on the top surface no closer than 1 mm to the adjacent indentations or the margin of the specimen, and were averaged to determine the hardness value for each specimen.

The compressive strength (CS) and diametral tensile strength (DTS) were calculated using the following equations.

$$\text{Compressive strength (CS)} = 4P/d^2$$

$$\text{Diametral tensile strength (DTS)} = 2P/dT$$

where **P** was the force (Newtons) applied, **d** (mm) the specimen diameter and **T** height of the specimen (mm).

The results of CS, DTS and VHN tests were statistically analysed using ANOVA. The post hoc analysis was performed using the Scheffé test considering storage media and time for comparisons within each material. The threshold value for statistical significance was set at a P value of 0.01.

## RESULTS

The compressive strengths for the three materials investigated are shown in Table 3. Fuji II LC and Fuji IX stored in all three media, resulted in modest changes at different testing times and an increase at 12 weeks. For Dyract EXTRA however, the compressive strength continuously decreased over 12 weeks. Statistical analysis failed to reveal any significant differences in the effects of storage solutions for each individual material ( $P>0.01$ ). Considering time as a factor revealed some significant differences for Fuji IX and Dyract EXTRA as shown in the table.

Table 4 shows the results of the diametral tensile strength. Similar pattern of change was evident for Fuji II LC and Fuji IX, where gradual changes resulted in an overall increase

**Table 4.** *Diametral tensile strength of materials tested.*

Material/ Media	24hr		1w		4w		12w	
<i>FujiII LC</i>								
Water	27.66 (2.59)	a	27.86 (2.83)	a	33.78 (2.94)	b	36.03 (1.78)	b
Saliva	27.11 (2.34)	a	26.81 (3.53)	a	36.07 (2.63)	b	37.89 (3.37)	b
SL	26.73 (2.09)	a	31.37 (2.89)	ab	35.01 (2.49)	b	35.60 (4.02)	b
Average	27.17 (2.26)	a	28.68 (3.55)	a	34.96 (2.73)	b	36.50 (3.20)	b
<i>FujiIX</i>								
Water	25.03 (1.44)	a	27.60 (2.81)	a	29.70 (4.22)	a	38.20 (3.82)	b
Saliva	25.99 (1.64)	a	32.18 (2.83)	b	31.55 (3.71)	b	34.80 (4.28)	b
SL	25.33 (1.80)	a	28.87 (3.49)	ab	32.41 (2.95)	bc	35.03 (2.83)	c
Average	25.45 (1.60)	a	29.55 (3.52)	b	31.21 (3.66)	b	36.01 (3.74)	c
<i>Dyract EXTRA</i>								
Water	43.51 (3.43)	a	37.29 (3.46)	b	33.91 (2.76)	bc	31.70 (2.18)	c
Saliva	41.07 (2.11)	a	37.09 (4.31)	ab	32.81 (1.69)	bc	29.49 (2.45)	c
SL	40.06 (2.36)	a	34.76 (2.60)	b	33.58 (3.19)	b	28.83 (3.16)	c
Average	41.55 (2.96)	a	36.38 (3.55)	b	33.43 (2.53)	b	29.92 (2.80)	c

*Mean values sharing the same lower case letters were not statistically different for comparisons within each row ( $P>0.01$ ). No statistically significant differences when comparing values within a column for each material ( $P>0.01$ ).*

at 12 weeks. This is in contrast with Dyract EXTRA which showed continuous decreases in diametral tensile strength up to the last testing point. The comparison by solution for each material failed to reveal any statistically significant differences ( $P>0.01$ ). Considering time as a factor revealed some significant changes as shown in the table.

Table 5 shows the results of Vickers hardness test. For each material, regardless of the storage media, similar pattern of change was evident over the testing period. Comparison by storage media for each material at each testing point revealed some statistically significant results as shown in the table.

## DISCUSSION

Studies in the past have revealed that ionic and polymeric restorative materials such as glass ionomers, composite resins and hybrid systems interact with various aqueous media<sup>1,11-13</sup>. In addition to the effects associated with storage in water alone, saliva ions, pH levels and alcohol contents of the storage media are important in initiating interactions which may affect physical properties of the restorative materials<sup>11,13,14</sup>.

The aim of this *in vitro* study was to investigate possible changes in compressive strength, diametral tensile strength (CS and DTS) and Vickers hardness (VHN) of a conventional glass ionomer (Fuji IX), a resin modified glass ionomer (Fuji II LC), and a compomer material (Dyract EXTRA) following storage in water and artificial saliva with and without intermittent exposure to Listerine.

Compressive and diametral tensile strength can be indicative of a restorative materials' resistance to forces generated during function<sup>15</sup>. Vickers hardness of a material is resistance to indentation or penetration<sup>16</sup> and in clinical terms it may indicate materials' wear resistance and their ability to maintain form stability<sup>17</sup>. It has been suggested in the past that resin modified glass ionomers (RM GICs) have improved mechanical properties compared to the conventional ones<sup>18</sup>. This has been particularly true regarding their flexural strength<sup>18,19</sup>. However, when comparing other physical properties such as CS and VHN, the results have proved that this may not be the case<sup>20</sup>. The results of our study showed that Fuji IX and Fuji II LC despite their different characteristics, composition and setting reactions, had similar CS and DTS values at 24 hr and throughout the period of this study. In a study by Yli-urpo et al<sup>20</sup>,

**Table 5.** *Vickers hardness values of materials tested.*

Material/ Media	24hr	1w	4w	12w
<i>FujiII LC</i>				
Water	60.49 AB, a (3.94)	64.17 A, a (5.43)	82.34 b (4.49)	78.97 A, b (7.99)
Saliva	63.73 A, a (5.10)	69.46 B, b (4.99)	80.61 c (5.28)	79.35 A, c (5.40)
SL	58.50 B, a (3.43)	59.01 C, a (4.27)	84.38 b (4.61)	72.04 B, c (5.48)
Average	60.97 a (4.71)	64.21 b (6.49)	82.44 c (4.99)	76.79 d (7.17)
<i>FujiIX</i>				
Water	80.81 AB, a (7.43)	58.40 A, b (4.96)	100.04 A, c (6.93)	81.43 AB, a (8.04)
Saliva	84.29 A, a (9.65)	75.18 B, b (4.79)	102.40 A, c (4.97)	75.94 A, b (6.38)
SL	76.74 B, a (8.77)	63.42 C, b (7.53)	86.66 B, c (8.04)	85.04 B, c (6.32)
Average	80.61 a (9.11)	65.67 b (9.17)	96.36 c (9.66)	80.80 a (7.84)
<i>Dyract EXTRA</i>				
Water	66.73 A, a (7.41)	57.49 b (5.26)	55.68 A, b (5.29)	72.95 c (6.50)
Saliva	56.85 B, a (7.08)	55.41 a (6.81)	62.02 B, b (6.06)	69.90 c (5.67)
SL	68.30 A, a (6.76)	58.59 b (6.03)	54.61 A, b (3.99)	68.08 a (5.45)
Average	63.96 a (8.66)	57.16 b (6.14)	57.44 b (6.09)	70.31 c (6.16)

*Values sharing the same lower case letters were not statistically different for comparisons within each row (P>0.01). Values sharing the same upper case letter were not statistically significant for comparisons within a column for each material (P>0.01).*

similar findings related to CS were reported when comparing conventional and RM GIC after a 180 day period of storage in water.

Earlier studies have suggested that the CS of some conventional and RM GICs tend to increase with prolonged storage in water<sup>21,22</sup>. The results of our study support this finding. A similar trend for DTS was observed. Irrespective of the storage media, CS and DTS increased continuously for Fuji II LC and Fuji IX during the period of this study.

Dyract EXTRA behaved in a considerably different way during the period of our study. CS and DTS values at 24 hr were significantly higher for Dyract EXTRA compared to the other two materials. However, the values for both test continuously decreased for Dyract EXTRA irrespective of the storage media. An explanation for this different behaviour could be the fact that Dyract EXTRA belongs to a different family of restorative materials. Physical properties of a hybrid material is very much dependant on how close their formulations are to glass ionomer or composite resin family. In other words how much polyacid or resin components they contain<sup>23</sup>. Improvement in strength of Fuji II LC and Fuji IX with time may be related to maturation state and their continued setting reaction<sup>24</sup>. Whereas for

Dyract EXTRA storage in aqueous media may have resulted in dissolution of water soluble substances, causing degradation of components and ultimately decreasing strength of the material<sup>1</sup>. For all three materials, effects of time were proved to be more significant than the storage media.

In other words, the significant changes in the mechanical properties (CS and DTS) of the materials investigated occurred by prolonged exposure regardless of the storage media. This is an interesting finding which is contrary to some previous results in other studies<sup>9</sup>.

Listerine is a mouthwash with high alcohol concentration and a low pH. The high alcohol content could cause dissolution of the inorganic contents of the polymeric materials and the low pH facilitates ionic exchanges with the poly-acid based materials. Our results indicate that despite possible chemical interactions between the saliva ions /Listerine and restorative materials, the tests employed in this study, failed to detect any possible effects of these interactions on the physical properties of the materials investigated in the period of the study. This may be related to the fact that the exposure regime employed here, was mild compared to other *in vitro* studies. It is important to bear in mind the period of this study is short relative to

the life expectancy of the restorative materials in a clinical situation. Different results may be achieved in more prolonged exposure periods.

Vickers hardness testing revealed a unique pattern of change in hardness for each material over time regardless of the storage media. Comparisons by the storage media could not be conclusive as the results were not consistent at different testing points. This may be a result of cyclic changes at the surface with periods of dissolution of the matrix and collapse of the structure followed by exposure of a fresh layer. The inconsistent pattern may also partly be related to lack of enough sensitivity related to VHN testing which allows for a relatively large margin of error for the operator when measuring the diameter of indentations on the surface. Further testing using different methods may be able to produce more conclusive results.

## CONCLUSIONS

1. Physical properties of Fuji II LC and Fuji IX can improve over a period of few weeks storage in water and artificial saliva.
2. In the same period Dyract EXTRA shows some deterioration in physical properties.
3. Exposure to Listerine for the regime recommended for clinical use does not seem to affect the strength of the materials investigated in this study.
4. Consideration should be given to the choice of dental materials by the dental professionals in relation to mechanical demands of the restorations and the period they are expected to be in service.

## ADDRESS FOR CORRESPONDENCE

Leili Sadaghiani, E-mail: sadaghianil@cf.ac.uk

## REFERENCES

1. Nicholson, J.W. and Alsarheed, M. Changes on storage of polyacid-modified composite resins. *J. Oral Rehab.*, 1998; **25**:616-20.
2. Cattani-Lorente, M.A., Dupuis, V., Payan, J., Moya, F. and Meyer, J.M. Effect of water on the physical properties of resin-modified glass ionomer cements. *Dent. Mater.*, 1999; **15**:71-8.
3. Cattani-Lorente, M.A., Dupuis, V., Moya, F., Payan, J. and Meyer, J.M. Comparative study of the physical properties of a polyacid-modified composite resin and a resin-modified glass ionomer cement. *Dent. Mater.*, 1999; **15**:21-32.
4. Nicholson, J., Anstice, H.M., and McLean, J.W. A preliminary report on the effect of storage in water on the properties of commercial light-cured glass-ionomer cements. *B.D.J.*, 1992; **173**:98-101.
5. Adusei, G.O., Deb, S. and Nicholson, J.W. A comparison of some properties of contemporary polyacid-modified composite resins ("compomers") for use in clinical dentistry. *Dental Forum Poland*, 2004; **2**:19-24.
6. Attin, T., Vataschki, M. and Hellwig, E. Properties of resin-modified glass-ionomer restorative materials and two polyacid-modified resin composite materials. *Quintessence Int.*, 1996; **27**:203-9.
7. Zimmer, S., Kolbe, C., Kaiser, G., Krage, T., Ommerborn, M. and Barthel C. Clinical efficacy of flossing versus use of antimicrobial rinses. *J. Periodontol.*, 2006; **77**:1380-5.
8. Ciancio, S. Improving oral health: current considerations. *J. Clin. Periodontol.*, 2003; **30** Suppl **5**:4-6.
9. Sadaghiani, L., Wilson, M.A. and Wilson, N.H.F. Effect of selected mouthwashes on the surface roughness of resin modified glass-ionomer restorative materials. *Dent. Mater.*, 2007; **23**:325-34.
10. Fusayama, T., Katayori, T. and Nomoto, S. Corrosion of gold and amalgam placed in contact with each other. *J. Dent. Res.*, 1963; **42**:1183-97 cited in Kaplan, A.E., et al. Effects of variation in particle size on biaxial flexural strength of two conventional glass ionomer cements. *J. Oral. Rehab.*, 2004; **31**:373-8
11. Okada, K., Tosaki, S., Hirota, K. and Hume WR. Surface hardness change of restorative materials stored in saliva. *Dent. Mater.*, 2001; **17**: 34-39.
12. Czarnecka, B., Limanowska-Shaw, H. and Nicholson J.W. Buffering and ion-release by a glass-ionomer cement under near-neutral and acidic conditions. *Biomater.*, 2002; **23**:2783-8.
13. Algera, T.J., Kleverlaan, C.J., Prahl-Andersen, B. and Feilzer A.J. The influence of environmental conditions on the material properties of setting glass-ionomer cements. *Dent. Mater.*, 2006; **22**:852-856.
14. Aguiar, F.H., Braceiro, A.T., Ambrosano, G.M. and Lovadino, J.R. Hardness and diametral tensile strength of a hybrid composite resin polymerised with different modes and immersed in ethanol or distilled water media. *Dent. Mater.*, 2005; **21**:1098-103
15. Cobb, D.S., Vargas, M.A. and Rundle, T. Physical properties of composites cured with conventional light or argon laser. *Am. J. Dent.*, 1996; **9**:199-202
16. O'Brein, W.J. *Dental Materials and Their Selection*, 2<sup>nd</sup> edn. Quintessence Co Inc, IL,USA. 1997;13.
17. Baharav, H., Abraham, D., Cardash, H.S. and Helft M. Effect of exposure time on the depth of polymerization of a visible light-cured composite resin. *J. Oral Rehab.*, 1998; **15**:167-72.
18. Wilson, A.D. Resin-modified glass-ionomer cements. *Int. J. Prosthodont.*, 1990; **3**:425-9.
19. Xie, D., Brantley, W.A., Culbertson, B.M. and Wang, G. Mechanical properties and microstructures of glass-ionomer cements. *Dent. Mater.*, 2000; **16**:129-38
20. Yli-Urpo, H., Lassila, L.V., Narhi, T. and Vallittu, P.K. Compressive strength and surface characterization of glass ionomer cements modified by particles of bioactive glass. *Dent. Mater.*, 2005; **21**:201-9.
21. Williams, J.A., Billington, R.W. Changes in compressive strength of glass-ionomer restorative materials with respect to time periods of 24 h to 4 months. *J. Oral Rehab.*, 1991; **18**:163-8.
22. Mitra, S.B. and Kedrowski, B.L. Long-term mechanical properties of glass ionomers. *Dent. Mater.*, 1994; **10**:78-82.
23. Mendonca, J.S., Souza, M.H. Jr and Carvalho, R.M. Effects of storage time on microtensile strength of polyacid-modified resin composites. *Dent. Mater.*, 2003; **19**:308-12.
24. Ellakuria, J., Triana, R., Minguez, N., Soler, I., Ibaseta G, Maza, J. and Garcia-Godoy, F. Effect of one-year storage on the surface microhardness of resin-modified versus conventional glass-ionomer cements. *Dent. Mater.*, 2003; **19**:286-90.