Research article

Staining effect of various beverages on and surface nano-hardness of a resin coated and a non-coated fluoride releasing tooth-coloured restorative material: An in-vitro study

R. Kurinji Amalavathy, Hrudi Sundar Sahoo*, Sushmita Shivanna, Jayanthi Lingaraj, S. Aravinthan
Department of Conservative Dentistry and Endodontics, Sathyabama Dental College and Hospital, Chennai, India

ARTICLE INFO
Keywords:
Dentistry
Staining
Beverages
Hardness
Spectrophotometry
Color
Glass ionomer

ABSTRACT
Objective: The objective of this study was to compare staining effect of various beverages on and surface nano-hardness of a resin coated and a non-coated fluoride releasing tooth-coloured restorative material.

Materials and methods: Two restorative materials, Equia Forte Fil (coated with Equia Forte Coat) and Cention N (non-coated), were evaluated in this study. Fifty samples (n = 50) were prepared from each restorative material. Ten samples of each material were randomly placed in Phosphate Buffer Saline (PBS) solution, Tea, Coffee and Coke solutions. Spectrophotometer readings were recorded after 24 hours of sample preparation followed by 7th, 14th, 21st and 28th day. Colour difference (ΔE) for each sample was calculated at the specified time intervals. The remaining ten samples of each restorative material were subjected to nano-hardness testing. Nano-hardness values at 1st day and 28th day were noted. Colour difference and nano-hardness values were statistically analysed.

Results: By the 28th day, all samples from the solutions were stained. Equia Forte Fil coated with Equia Forte Coat samples showed better resistant to staining solutions than Cention N samples. Surface nano-hardness of both the materials increased from the 1st day to 28th day and were comparable.

Conclusion: Over a period of time, changes in colour stability and nano-hardness, are inevitable. Cention N showed more potential to undergo surface discolouration than Equia Forte Fil. Both materials exhibited increased nano-hardness over a period of time. Cention N exhibited higher surface nano-hardness when compared to Equia Forte, by the 28th day.

Clinical significance: Resin coated, high viscous and fluoride releasing glass ionomers can delay the staining effect by various beverages and exhibit better initial surface nano-hardness when compared to a non-coated alka-site based, fluoride releasing restoration.

1. Introduction

Glass ionomer cements (GIC) have been used in restoration of primary dentition or in small “non-load” fillings in the permanent dentition. Recent researches concentrate on the development of restorative materials that can overcome the shortcomings of resin composites [1]. Existing literature has shown that the high viscous glass ionomer cement is not inferior to dental amalgam restorations [2,3]. High viscous glass ionomer cement with a resin coating in posterior restorations can be a success due to ease of use, relative tolerance to moisture, anti-cariogenic nature, and fluoride release [4]. Unlike conventional glass ionomers, the resin modified glass ionomers exhibit superior wear resistance [5].

Equia Forte Fil (GC America INC) is a recent addition to the high viscosity GIC with resin coating. It is a fast setting, self-adhesive, and aesthetic restorative material. It is a metomorphic glass formed by ultrafine highly reactive glass dispersed within the glass ionomer fillers. It has a strong matrix structure that contributes to improved physical properties, wear resistance, and fluoride release. EQUIA Forte Coat is a light cured, cross-linking monomer. It provides a protective, smooth and sealed surface with remarkable strength. It also improves wear resistance, translucency, and aesthetics. EQUIA Forte Fil is available in the following Vita shades: A1, A2, A3, A3.5, B1, B2, B3 and C4.

Cention N (Ivoclar Vivadent) is a resin based, self-adhesive, and self-curing restorative material. It is also a bulk fill tooth-coloured restorative...
material that belongs to a new category of filling material called “Alkasil”. This material uses alkaline filler, capable of releasing acid-neutralizing ions and fluorides. It is available in A2 shade. Apart from aesthetic considerations, surface hardness plays a major role in determining the longevity of posterior restorative materials. Currently, both Equia Forte Fil and Cention N are used for restorations of class I, II, and cervical defects in permanent teeth. The current literature is devoid of data with respect to the colour stability and surface nano-hardness of these newer advanced, fluoride releasing restorative materials. Hence, the aim of this study was to compare staining effect of various beverages and evaluate the nano-hardness of these posterior tooth-coloured restorative materials.

The following null hypotheses were tested: (a) Both the materials were not stained by beverages; and (b) Both the materials had similar surface nano-hardness.

2. Materials and methods

2.1. Sample preparation

A2 shade of Equia Forte (Fuji, GC) and Cention N (Ivoclar) were used in this study. Following the manufacturer’s instruction, fifty samples were prepared for each of these materials and placed inside a metal ring. The metal ring was placed between two glass slabs for the removal of excess material [6]. All samples measured 10 mm in diameter and 4 mm in thickness. After the initial set, all samples were finished and polished. Polishing was done by using coarse, medium, fine, and superfine Soflex discs in sequence (Soflex system 3M, US). All Equia Forte samples received a layer of Equia Forte Coat and light cured using Coltulux (Coltene/Whaledent, USA) at 1400mW per-cm² for 40 s at a distance of 2mm perpendicular to the sample surface using a light shield attached to the tip of handpiece. All samples were incubated at 37 degrees centigrade for 24 h. Each sample was placed on a black ground. The base-line L, a, and b values were recorded using a digital spectrophotometer (Vita EasyShade4.0, VITA, USA). As per the manufacturer’s instructions, the spectrophotometer was manually calibrated before each set of measurements.

2.2. Staining solutions

Coffee, tea, coke, and phosphate buffered saline (PBS) solution were used in this study (Table 1). Ten samples from each of the experimental materials were stored in these solutions and incubated at room temperature. Solutions were replaced every 24 hours at room temperature for a test period of 28 days. The CIE 1976 L* a*b* colour system was used to compute the colour differences. The following formula was used to calculate the colour differences (ΔE) at various time intervals: ΔE = [(ΔL)² + (Δa)² + (Δb)²]¹/²

Colour differences were compared among the samples obtained from all staining solutions.

2.3. Nano-hardness

Surface nano-hardness values of 10 samples of each restorative material were calculated after 24 hours and 28 days of storage in PBS solution. Fresh PBS solution replacement was carried out at every 24 hours. The HYSITRON T1 950 Tribo Indenter with the Berkovich diamond indenter tip was used for nano-hardness measurements. The tip was calibrated with fused quartz sample. This machine load resolution is less than 1 Nano-N and displacement resolution 0.04 NM. In this study, the load was held for 10 s starting from 500 μN, 1000 μN, 1500 μN and finally 2000 μN. For a particular load, 5 average indentations were recorded to increase the reliability of the results. The Oliver -Pharr method [7] was used to calculate the nano-hardness values.

2.4. Statistical analysis

The colour difference (ΔE) of samples from each solution, were analyzed by non-parametric Friedman’s ANOVA with repeated measures. At every time interval, the colour difference between the samples from different staining solutions was analyzed by the Kruskall-Wallis test and Post-hoc Dunn test. The final colour differences between the materials were analyzed using the Mann-Whitney U test. All tests were performed under a confidence level of 95% (p < 0.05).

The mean nano-hardness values of each restorative material were calculated after 24 hours and 28th day and were compared using the Wilcoxon Signed Rank test, whereas the final mean nano-hardness of both the materials were compared with the Kruskal Wallis test.

3. Results

All samples showed visible (ΔE>3) colour change by the 28th day irrespective of the staining solutions. The mean ΔE values and standard deviations of all Equia Forte Fil samples and Cention N samples are mentioned in Table 2 and Table 3, and shown in Figure 1 and Figure 2, respectively. Upon testing the samples for surface nano-hardness, there was a significant difference (p < 0.05) in between 1st day and 28th day as mentioned in Table 4.

4. Discussion

The longevity of posterior amalgam restorations done with optimum condensation forces is very high. But the poor aesthetic appeal and mercury content was always a concern. This has led to a surge of tooth-coloured restorative materials in recent years. Colour stability is an important pre-requisite for considering a tooth-coloured restorative material. Many patients insist on replacing a stained restoration, irrespective of its location inside the oral cavity [19]. Hence, it is necessary to investigate the colour stability of such restorative materials.

To avoid inherent errors in manual colour evaluation, VITA Easy Shade (digital spectrophotometer) was employed to record the CIE L* a*b* values [8]. The ΔE mean values of samples in each staining solution were compared at 7th, 14th, 21st and 28th day [9]. A ΔE value of >3, denotes a perceptible colour change [10]. Many studies have evaluated the colour stability of various aesthetic restorations [6,11,12,13]. These studies have concluded that beverages such as tea, coffee and coke, stain the restorations over a period of time.

According to Guler and others [14], 15 days of storage in a coffee solution simulated coffee consumption for 1 year by an individual. In this study, 28 days of storage were followed to simulate approximately 20 months of beverage consumption. Thermocycling was avoided in this study to rule out its possible effects on the colour changes of the samples [15,16].

| SL No. | Solutions                                           | Manufacturer                                   |
|-------|-----------------------------------------------------|-----------------------------------------------|
| 1.    | PBS (Phosphate buffered saline- neutral pH)         | Sigma Aldrich, Buchs, Switzerland             |
| 2.    | Tea (2 Tea bags of 1.9g each mixed in 300 ml of boiling water for 10 min) | Instant tea bag, Taj Mahal tea house, Mumbai, India |
| 3.    | Coke                                                | Coca-cola, India                              |
| 4.    | Coffee (1 sachet of 3.1g mixed in 300 ml of boiling water for 10 min followed by filtration) | Instant Coffee sachet, Nescafe, Switzerland |
At the end of 28 days, all samples exhibited a ΔE value greater than 3.0. A rise in ΔE value from the 1st day till the 28th day in all samples, suggests a possible time-dependent deterioration of the samples’ surface. Such a phenomenon can be due to increased fluoride related ion-exchange activity at the surface of the samples on exposure to acidic pH of the beverages or the neutral pH 7 of the PBS solution [17].

On the 7th day, all the solutions visibly stained Equia Forte samples without any significant difference. At the end of 28 days, coffee and tea solutions stained Equia Forte samples more significantly than the rest of the solutions. Contrastingly, on the 7th day, there was a statistically significant difference between staining of Cention N samples obtained from all solutions. Among the solutions, PBS solution continued to stain the Cention N samples at minor perceptible levels from the 7th day to 28th day. By the 28th day, tea solution had predicted the most severe staining on the Cention N samples, though statistically not different from coffee solution. From the 7th day to 14th day Cention N samples showed some degree of resistance to staining in coffee solution. Thereafter, there was a perceptible colour change till 21st day followed by a negligible colour change till the 28th day. Cention N samples immersed in coke solution showed no perceptible colour changes from 7th day till the 21st day. From the 21st day to 28th day, a perceptible discolouration was evident on the 28th day.

At the end of 28 days, all samples exhibited a ΔE value greater than 3.0. A rise in ΔE value from the 1st day till the 28th day in all samples, suggests a possible time-dependent deterioration of the samples’ surface. Such a phenomenon can be due to increased fluoride related ion-exchange activity at the surface of the samples on exposure to acidic pH of the beverages or the neutral pH 7 of the PBS solution [17].

On the 7th day, all the solutions visibly stained Equia Forte samples without any significant difference. At the end of 28 days, coffee and tea solutions stained Equia Forte samples more significantly than the rest of the solutions. Contrastingly, on the 7th day, there was a statistically significant difference between staining of Cention N samples obtained from all solutions. Among the solutions, PBS solution continued to stain the Cention N at minor perceptible levels from the 7th day to 28th day. By the 28th day, tea solution had inflicted the most severe staining on the Cention N samples, though statistically not different from coffee solution. From the 7th day to 14th day Cention N samples showed some degree of resistance to staining in coffee solution. Thereafter, there was a perceptible colour change till 21st day followed by a negligible colour change till the 28th day. Cention N samples immersed in coke solution showed no perceptible colour changes from 7th day till the 21st day. From the 21st day to 28th day, a perceptible discolouration was evident on the 28th day.

On the whole, it was noteworthy that the ΔE values of Equia Forte samples were significantly lower than the ΔE values of Cention N samples. Such a phenomenon may be attributed to the presence of Equia Coat. As acclaimed by the manufacturer, Equia Coat is resistant to any degradation at low pH. Hence, the first null hypothesis (a) was rejected.

Surface nano-hardness values of both the materials were significantly different. It can be possibly due to the difference in their composition. Surface hardness is indirectly proportional to wear resistance. For an instance, materials with low surface hardness tend to wear off easily. Micro and macro surface hardness can be determined by Rockwell, Vicker, Brinell, and Knoop test methods. But nano-indentation test methods have yielded higher precision results [18]. At present, there are

### Table 2. ΔE values of Equia Forte.

| Solutions | 7th day | 14th day | 21st day | 28th day |
|-----------|---------|----------|----------|----------|
| PBS**     | 6.98 ± 2.28 | 7.74 ± 2.04 | 7.96 ± 1.83 | 9.66 ± 2.15 |
| COFFEE**  | 5.89 ± 2.27 | 7.13 ± 2.13 | 8.76 ± 2.79 | 12.09 ± 2.96 |
| TEA**     | 8.05 ± 1.99 | 8.30 ± 1.76 | 11.41 ± 1.16 | 11.53 ± 1.24 |
| COKE      | 5.69 ± 2.9420 | 5.71 ± 2.0836 | 6.03 ± 1.7487 | 6.23 ± 1.7231 |

The values are mean ± SD.

- **Significant values in Kruskal-Wallis test.
- † Significant values in Friedman’s ANOVA for repeated measures.
- ‡ Significant values in Mann Whitney U test.

### Table 3. ΔE values of Cention N.

| Solutions | 7th day | 14th day | 21st day | 28th day |
|-----------|---------|----------|----------|----------|
| PBS**     | 3.85 ± 2.36 | 5.76 ± 2.71 | 7.44 ± 2.47 | 8.65 ± 2.22 |
| COFFEE**  | 16.45 ± 2.46 | 18.42 ± 3.21 | 27.10 ± 2.56 | 28.99 ± 2.32 |
| TEA**     | 20.19 ± 2.90 | 34.65 ± 2.01 | 35.37 ± 2.61 | 35.77 ± 2.55 |
| COKE**    | 5.68 ± 1.60 | 7.22 ± 1.37 | 9.54 ± 2.50 | 18.68 ± 2.31 |

The values are mean ± SD.

- **Significant values in Kruskal-Wallis test.
- † Significant values in Friedman’s ANOVA for repeated measures.
- ‡ Significant values in Mann Whitney U test.

---

**Figure 1.** Mean delta E values of Equia Forte samples at different time intervals in different staining solutions.
a limited number of studies relevant to the nano-mechanical properties of enamel and dentine [19]. Due to heterogeneity in testing environment, it was not possible to co-relate the nano-hardness values of Equia Forte and Cention N (in this study), with enamel and dentine. Hence, future studies are necessary for formulating a standardized testing environment.

The surface nano-hardness values of Equia Forte Fil samples were higher compared to Cention N samples on the first day. The reason for a high initial nano-hardness can be attributed to the application of Equia Coat. According to the manufacturer, the Equia coat laminates and toughens the underlying restoration. But, the final surface nano-hardness of Cention N was higher than Equia Forte. Hence, Cention N showed significant wear resistance improvement over 28 days. Thus, the second null hypothesis (b) was also rejected. Future researches are required to find out a reason for the above difference in final surface hardness.

5. Conclusions

Irrespective of the restorative material and their protective coating compositions, changes in colour stability and their surface nano-hardness is inevitable over a period of time. It was presumed that both of these fluoride releasing restorative materials demonstrated discernible staining in all beverages and should be avoided when aesthetics is of foremost significance. Between the two restorative materials, Cention N showed more potential to undergo surface discolouration than Equia Forte. Although both materials showed improved surface nano-hardness, Cention N exhibited higher nano-hardness at the end of 28 days.

Declarations

**Author contribution statement**

Kurinji Amalavathy R, Sushmita Shivanna: Conceived and designed the experiments; Wrote the paper.

Hrudi Sundar Sahoo: Conceived and designed the experiments; Analyzed and interpreted the data; Wrote the paper.

Jayanthi Lingaraj: Performed the experiments; Contributed reagents, materials, analysis tools or data.

Aravinthan S: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

**Funding statement**

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

**Competing interest statement**

The authors declare no conflict of interest.

**Additional information**

No additional information is available for this paper.

**References**

[1] P.E. Petersen, Ramon Baez, Stella Kwan, Hiroshi Ogawa, Future Use of Materials for Dental Restoration World Health Organization, WHO, Geneva, Switzerland, 2010. https://apps.who.int/iris/handle/10665/202500.

[2] S. Mickenauscht, V. Yengopal, Direct contra naïve-indirect comparison of clinical failure rates between high-viscosity GIC and conventional amalgam restorations: an empirical study, PloS One 8 (2013), e78397.

[3] S. Mickenautsch, V. Yengopal, Failure rate of high-viscosity GIC based ART compared with that of conventional amalgam restorations– evidence from an update of a systematic review, South Afr. Dent. J. 67 (2012) 329–331.

[4] A.M. Kielbasa, G. Glockner, M. Wolgen, R. Glockner, Systematic review on highly viscous glass-ionomer cement/resin coating restorations [Part I]: do they merge Minamata Convention and minimum intervention dentistry? Quintessence Int. 48 (2017) 9–18.

[5] A.U. Yap, J.C. Teo, S.H. Teoh, Comparative wear resistance of reinforced glass ionomer restorative materials, Oper. Dent. 26 (2001) 343–348.
[6] M.A. Vargas, H.L. Kirchner, A.M. Díaz-Arnold, V.L. Beck, Colour stability of ionomer and resin composite restoratives, Oper. Dent. 26 (2001) 166–171.

[7] W. Oliver, G. Pharr, An improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments, J. Mater. Res. 7 (1992) 1564–1583.

[8] M.H. Kalantari, S.A. Ghoraiishian, M. Mohaghegh, Evaluation of accuracy of shade selection using two spectrophotometer systems: Vita Easy shade and Degudent Shades pilot, Eur. J. Dermatol. 11 (2007) 196–200.

[9] R. Bagheri, M.F. Burrow, M. Tyas, Influence of food simulating solutions and surface finish on susceptibility to staining of aesthetic restorative materials, J. Dent. 33 (2005) 389–398.

[10] A.U. Yap, C.P. Sim, W.L. Loh, J.H. Teo, Human eye versus computerized colour matching, Oper. Dent. 24 (1999) 358–361.

[11] S. Inokoshi, M.F. Burrow, M. Kataumi, T. Yamada, T. Takatsu, Opacity and colour changes of tooth-coloured restorative materials, Oper. Dent. 21 (1996) 73–80.

[12] A. Leibrock, M. Rosentritt, R. Lang, M. Behr, G. Handel, Colour stability of visible light-curing hybrid composites, Eur. J. Prosthodont. Restor. Dent. 5 (1997) 125–130.

[13] Z. Ergucu, L.S. Turkun, A. Aladag, Colour stability of nano composites polished with one-step systems, Oper. Dent. 33 (2008) 413–420.

[14] A.U. Guler, F. Yilmaz, T. Kulunk, E. Guler, S. Kurt, Effects of different drinks on stainability of resin composite provisional restorative materials, J. Prosthet. Dent 94 (2005) 118–124.

[15] I. Gürdal, A. Atay, M. Eichberger, E. Cal, A. Üsümez, B. Stawarczyk, Colour change of CAD-CAM materials and composite resin cements after thermocycling, J. Prosthet. Dent 120 (2018) 546–552.

[16] Y.K. Lee, B.S. Lim, S.H. Rhee, H.G. Yang, J.M. Powers, Changes of optical properties of dental nano-filled resin composites after curing and thermocycling, J. Biomed. Mater. Res. 71B (2004) 16–21.

[17] A.A. Tag, A.K. Abdal, A.I. Dawood, The effect of pH on fluoride release of glass ionomer based restorative materials, Int. J. Dent. Sci. Res. 4 (2016) 52–57.

[18] M. Iijima, T. Muguruma, W.A. Brantley, S. Ito, T. Yusa, T. Saito, I. Masuguchi, Effect of bracket bonding on nanomechanical properties of enamel, Am. J. Orthod. Dentofacial Orthop. 138 (2010) 735–740.

[19] S.R. Braga, B.T. Vasconcelos, M.R. de Paula Macedo, V.R. Martins, M.A. Sobral, Reasons for placement and replacement of direct restorative materials in Brazil, Quintessence Int. 1 (4) (2007) 38.