ORIGINAL ARTICLE

Effect of Aging on Hardness of Glass Ionomer, Resin-Modified Glass Ionomer, Giomer and Compomer Dental Restorative Materials

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ABSTRACT

Objective: To assess deionized water's aging effect on the hardness of four direct tooth colored dental filling materials i.e., Glass Ionomer Cement, Resin Modified Glass Ionomer, Giomer and Compomer.

Study Design: An in vitro experimental study performed in triplicates.

Place and Duration of Study: The study was carried out at PG Laboratory, Department of Science of Dental Materials, Army Medical College, Rawalpindi, Pakistan from February 2023 to August 2023.

Methods: Three disc-shaped samples (n=3) of 10 mm diameter and 1 mm thickness of each restorative material were prepared using stainless steel molds per manufacturer's instructions. These disc-shaped samples were then suspended in a 15 mL conical centrifuge tube containing 10 mL deionized water, followed by incubation in a Forced Convection Laboratory Oven at 37°C where these were allowed to age over a period of 1-2 years. Vickers Hardness of each sample was then checked at two-time intervals, i.e., after 24 h setting and after 2.5 years of ageing through the Micro Vickers Hardness Tester calibrated at 1Kgf (9.80 N) with dwell time of 10 seconds and light intensity 10.

Results: After one day (24 h) aging in deionized water, maximum mean Vickers hardness number (VHN) was exhibited by Compomer (539.83±58.08 VHN) and least by GIC (175.75±24.47 VHN). After aging the sample for 912 days, maximum mean VHN was observed for GIC (420.67±99.66 VHN) and least by Compomer (354.33±9.22 VHN).

Conclusion: RMGIC sustains its hardness on aging, hardness of GIC increases while that of Giomer and Compomer decreases upon aging.

Keywords: *Aging, Compomer, Composite, Giomer, Glass Lonomer Cements, Hardness, Micro Indentation.*

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Introduction

Longevity of restorative materials in holistic oral environment is a crucial factor to be considered to ensure patient comfort and oral health. Mechanical properties of dental restoratives including hardness, wear resistance, compressive strength have a significant role to play in this account.[1] There are several approaches for estimating this quality, one of which is the Vickers micro hardness test.[2]

Tooth-colored dental filling materials like Glass Ionomer Cement (GIC), Resin Modified Glass Ionomer Cement (RMGIC) are used frequently, sometimes along with dental composite resins in various layering techniques.[3] Compomer and Giomer have also become very popular in restorative dentistry because of their distinctive characteristics such as fluoride release leading to prevention of caries, adequate biocompatibility but most of all, due to improved adhesion with tooth substrate.[4] These materials also possess coefficient of thermal expansion with values close to that of dentine.[5] As these materials are placed for long term use in oral cavity, these are subjected to continuous harsh exposure to factors like fatigue, corrosion, wear and erosion.[6] In such conditions, hardness plays a vital role in survival and longevity of such restorations.[7]

In the case of GICs, their inferior mechanical properties such as brittle nature, low hardness and wear resistance, low fracture resistance, reduced working time and moisture sensitivity limit their clinical application as a dental restorative material. GIC use for anterior teeth repair is more common where applied stresses are less than as posterior restorations.[8,9]

The development of RMGIC to prevail over the limitations of the mechanical properties of GIC has been very effective.[10,11] An esthetically superior restorative material called Giomer was introduced much later, which, like GIC has fluoroboroalumino silicate glass filler, with particle sizes ranging between 0.01-5µm engulfed in an organic resin matrix.[12] It has good applicability as direct anterior and posterior tooth restorative material and is frequently employed in restorative treatments for early childhood caries as well.[5] Modified Composites i.e., Compomers to which some components of GICs have been added, are also in use. Overall, the mechanical properties and aesthetics of Compomers are better than GICs but inferior to those of composites.[13]

A material's resistance to localized plastic deformation is measured by its hardness. Numerous hardness-testing methods, such as Knoop, Brinell, Vickers, and Rockwell, have been introduced over time, each with a unique scale.[14] However, the fundamental idea behind measuring hardness is to apply regulated loads while forcing an indenter into the surface under examination.[15] Vickers hardness number (VHN) is a substantial property that ensures resistance to plastic modifications and wear in brittle materials.[16] VHN is an excellent indicator of the longevity of GICs and other restorative materials.[11] However, as per the study by Kutuk et al., VHN of GIC was dependent upon type of GIC, type of treatment-protocols undergone during specimen preparation, type and duration of storage medium.[17]

A lot of work has been done on analyzing the short-term hardness of GICs; however, there has been a gap in analyzing the long-term effects of aging on the hardness of GICs, which is a more precise measure of wear resistance and durability of tooth-colored restorations. Hence, the objective of this study was to assess deionized water's aging effect on the hardness of four direct tooth colored dental filling materials, i.e., Glass Ionomer Cement, Resin Modified Glass Ionomer, Giomer and Compomer. Initial evaluation was conducted after 24 hours of setting and then later over 2.5 years during aging in deionized water. The null hypothesis was that aging does not affect VHN of the tested dental restorative materials.

Methods

The vitro study was carried out at PG Laboratory, Department of Science of Dental Materials, Army Medical College Rawalpindi, Pakistan from February 2023 to August 2023 after obtaining the approval from the institutional review committee of Army Medical College, Rawalpindi, Pakistan held on March 02, 2023 vide letter no ERC/ID/263. This study was designed to examine the VHN of four tooth-colored aged dental restorative materials; the descriptive details of the study groups are enlisted in table 1.

Table 1 Descriptive details of the study groups

S. No	Group (n=3)	Description			
		Company	Product Name	Shade	Lot No
1	Glass Ionomer Cement	GC Gold Label	HS Posterior EXTRA	-	1706011
2	Resin Modified Glass Ionomer Cement	GC Gold Label	Glass Ionomer Light – Cured Universal Restorative	-	1701141
3	Compomer	DENTSPLY	DyractR XP	A3	1603000759
4	Giomer	SHOFU INC	BEAUTIFIL II	A1	011713

Three disc-shaped samples (10 mm diameter and 1 mm thickness) of each dental restorative material were made using a stainless steel (SS) circular mold as per manufacturer's instructions. Samples of GIC were made by mixing powder and liquid in a ratio of 3:1 with setting time of 2-3 minutes. The other three dental restorative materials RMGIC, Compomer and Giomer, being light activated materials, were cured using LED light for sample preparation.

Each sample was suspended in a 15 mL conical centrifuge tube containing 10 mL deionized water via dental floss which was incorporated in each sample during its fabrication. In a test tube holding tray, all the tubes containing the test samples were kept in a Forced Convection Laboratory Oven–ESCO – (Singapore) – OFA-54-8 (Isotherm; Model: OFA-32-8, OFA-54-8, OFA-110-8, OFA-170-8, OFA-240-8) at 37°C where they were allowed to age over a period of 2.5 years. During aging the deionized water was replaced at regular intervals i.e., 1st day, 3rd day, 5th day, 7th day, 9th day, 14th day, 30th day and then after every 30th day till 2.5 years.

Vickers hardness number of each sample was obtained at two-time intervals i.e., after 24 h setting and after 2.5 years ageing through the Micro Vickers Hardness Tester calibrated at 1Kgf (9.80 N) with dwell time of 10 seconds and light intensity 10. On each sample surface, after locating the surface of the sample with the help of 10^{x} Objective lens, the indenter was turned to the front position for applying test force as per calibration. The 40^{x} Objective lens was then used to locate the diamond-shaped indentation on the sample surface. After measuring the diagonal of length (d1), the eyepiece was turned by 90° to measure another diagonal line length (d2). The d1 and d2 values were then use to obtain VHN values, which were then recorded. By employing, a similar method, four readings for each sample were taken, i.e., two per surface of each disc (n=12).

The collected data was then analyzed using SPSS version 29. The descriptive data, i.e., Mean and SD, were shown in a bar graph. Statistically significant differences in hardness of the tested dental restorative materials within and between groups at two-time intervals were calculated by two-way ANOVA and post hoc Tukey test, where *p*-value ≤0.05 was taken as significant.

Results

After one day (24 h) of aging in deionized water, the maximum mean VHN was exhibited by Compomer (539.83±58.08) and least by GIC (175.75±24.47). After aging the sample for 912 days, the maximum mean VHN was observed for GIC (420.67±99.66) and least by Compomer (354.33±9.22). However, GIC showed an increase in hardness upon aging, unlike Compomer and Giomer, which exhibited a fall in VHN upon aging. However, in Compomers, the decline was drastic when compared with Giomer. RMGIC maintained its hardness when subjected to aging, reporting only a slight increase in VHN. In Figure 1 where mean and standard deviation of VHN of all study groups at the planned two-time intervals are presented.

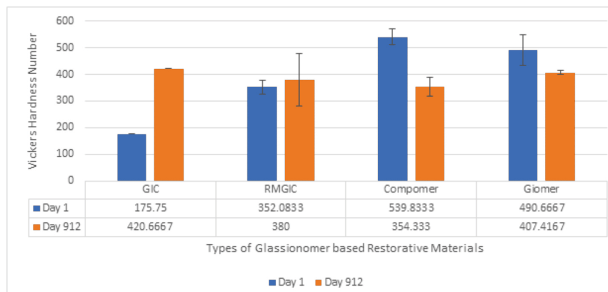

Fig 1. Vicker's Hardness Numbers (Mean & Standard Deviation) of the experimental dental restorative materials

A statistically significant difference in VHN of all the experimental dental restorative materials upon aging in deionized water were observed within and between the study groups *(p* value ≤0.001*)* as shown in Appendix 1 and Appendix 2. Pair-wise comparison showed a statistically significant difference in VHN between all the experimental groups *(p-value* ≤0.001*)* except between Compomer and Giomer *(p* value= .999*)* upon aging in deionized water as shown in Appendix 3.

Discussion

Dental restorative materials are under constant mechanical and thermal stresses in the oral cavity throughout their service life. During mastication these dental restoratives not only repeatedly occlude with opposing natural teeth but also are involved in grinding coarse foods of variable hardness. Due to this reason, these dental

Appendix 1: Tests of Within-Subjects Contrasts

Measure: Hardness

Source	Time Interval	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared	Noncent. Parameter	Observed Power[a]
Time Interval	Linear	25.010	1	25.010	.011	.917	.000	.011	.051
Time Interval * Group	Linear	612600.948	3	204200.316	88.616	.000	.858	265.848	1.000
Error(Time Interval)	Linear	101390.542	44	2304.330					

a. Computed using alpha = .05

Appendix 2: Tests of Between-Subjects Effects

Measure: Hardness,
Transformed Variable: Average

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared	Noncent. Parameter	Observed Power[a]
Intercept	14608620.844	1	14608620.844	5615.499	.000	.992	5615.499	1.000
Group	377858.448	3	125952.816	48.416	.000	.768	145.247	1.000
Error	114465.208	44	2601.482					

a. Computed using alpha = .05

Appendix 3: Post hoc Tests Multiple Comparisons

Measure: Hardness
Tukey HSD

(I) Group	(J) Group	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval Lower Bound	Upper Bound
GIC	RMGIC	-67.8333*	14.72380	.000	-107.1460	-28.5207
	Compomer	-148.8750*	14.72380	.000	-188.1876	-109.5624
	Giomer	-150.8333*	14.72380	.000	-190.1460	-111.5207
RMGIC	Compomer	-81.0417*	14.72380	.000	-120.3543	-41.7290
	Giomer	-83.0000*	14.72380	.000	-122.3126	-43.6874
Compomer	Giomer	-1.9583	14.72380	.999	-41.2710	37.3543

Based on observed means
The error term is Mean Square(Error) = 1300.741.
**. The mean difference is significant at the .05 level.*

restorative materials should retain adequate hardness to prevent abrasion and wear of dental restorative during service to maintain the restored tooth morphology and function for a longer duration. Hardness is a property that describes about the nature of a material, and its ability to resist abrasion and wear of material. Hardness testing is recommended to evaluate the strength and wear resistance of the material.[18]

Numerous material advancements in GICs as dental restoratives have led to moisture tolerance, easy handling, and chemical adhesion to tooth structure in addition to anti-bacterial and anti-cariogenic properties due to effective fluoride release. Unfortunately, GIC restoration exhibit poor esthetics, insufficient strength under bending and compressive forces, and poor wear resistance.[19] The wear rate of conventional GICs is 5X higher than amalgam and 3X greater than composite-resin dental restorative materials[20] which has limited its usage as temporary restorations. However, with numerous advances, GIC restorations demonstrate promising outcomes even in posterior stress-bearing sites of the oral cavity.[21] Reinforcement of conventional GIC with various materials like hydroxyapatite, silica, and zirconium oxide micro and nanoparticles lead to improvement in the mechanical properties.[8,9]

A short-term comparative study has been conducted in the past by Yap (2002), using depth sensing micro-indentation test on some of the direct tooth colored restorative materials. The materials being stored in distilled for 30 days at 37°C showed that Giomers had the highest value of hardness (72.5C ± 3.82), followed by highly viscous GIC (56.02 ± 10.70). Compomers showed hardness somewhat near to

GIC, slightly lower though (54.98 ± 5.83), while lowest hardness was exhibited by RMGIC (48.44 ± 7.11).[22] Similar findings were reported in this study for Giomer and GIC.

As per present study, GIC exhibited the lowest VHN after 24 h (175.75±24.47), however, its VHN reached to a maximum value after 912 days (420.67±99.66) showing that increase in hardness will ultimately lead to highest wear resistance potential of GIC upon further aging. The initial hardness of RMGIC was not high on day 1 (352.08±29.4 VHN), which was maintained with only a slight increase upon ageing after 912 days (380.00±36.12 VHN). The increase in hardness of GIC may be attributed to maturation of the acid-base reaction.[23] The initial low hardness value of GIC can lead to early failure of restoration, if care of ensuring a hydrophobic environment is not taken during early stages of setting.[24] The formation of GIC basically involves an acid base reaction followed by a continuous crosslinking process which is evident by the improvement in mechanical properties of the material over time.[25] For RMGIC, the reason of sustained VHN values may be accredited to its modified surface layer, protecting glass core in a hydrophilic environment.[26] However, Compomer and Giomer exhibited decline in VHN upon aging unlike GIC and RMGIC, indicating comparatively poor potential of wear resistance in oral environment contrasting to GIC and RMGIC. Compomers are basically polyacid modified composites in which usual filler is replaced by ion-leachable glass. Unlike glass ionomer products, Compors lack acid-base glass ionomer reaction which marks the chief difference between the two dental filling materials.[26]

Compomers loses its mechanical properties including hardness upon aging which makes it more prone to wear over a period of time in oral cavity.[13] This in accordance with this study findings where Compomers exhibited highest initial VHN (539.83±58.08) among the study groups initially, however, the Compomer's VHN (354±9.22) dropped more as compared to other study groups when tested on day 912. Absence of acid-base reaction and moisture absorption at slow rate by Compomer can possibly be an explanation for the drastic decline in its VHN values upon aging. Studies report reduction in mechanical strength of Compomers after several weeks of use, however, acceptable performance of Compomers in clinical studies in various applications marks its suitability in vivo.[13]

Giomers exhibited second highest initial VHN after 24 h (490.67±56.19), and increased VHN upon aging after 912 days (407.42±17.5) which make them ideal in terms of hardness and wear resistance in clinical usage as compared to GIC. This is in accordance with the study by Ilie and Stawarczyk which quoted that Giomer reached maximum hardness within 3 months and sustained it to a year 67.2 ± 11.55-N/mm^2, which was greater than GIC having a value of 61.4 ± 7.25-N/mm^2.[27]

Giomers are similar to Compomers except that in Giomers the acid-base reaction is completed prior to combining the filler with resin. Pre-reacted glass polyalkenoate complex is formed by reaction of the aluminosilicate glass with the polyacid.[27] This can again be a presumed explanation for a moderate effect on VHN values which decreased upon aging. Another study by Söderholm, Zigan suggests that the presence of pre-polymerized fillers in Giomer may also contribute to this effect.[28] This phenomenon was described by the study conducted by Welker et al., which stated that Giomer reported high hardness numbers upon aging as these are highly filled materials when compared with Compomers which have less density of fillers. Hence indicating a direct proportionality of hardness with filler content.[29] Similar results have been reported for dental composites which showed a decline in Knoop hardness values when exposed to artificial aging.[30]

Similar to the present study, Vijyan et al studied the effect of aging on tooth-colored material but the aging time in distilled water was short i.e., 23 h and reported somewhat similar findings as the first interval of this study i.e., Giomers upon 23 h aging in distilled water exhibited highest VHN (53.833±0.965 VHN) followed by hybrid composites (52.450±0.689 VHN) then RMGIC(50.77±0.633 VHN) but Compomers reported least hardness (40.667±0.812 VHN).[31]

Up till now no study is available on ageing of such tooth-colored dental restorative materials for such a long time which gives this study an edge as it is clinically more relevant considering the service life of these restorative materials upon placement in oral environment. The limitation of this study is that microhardness was calculated at only two-time intervals instead of multiple time points and secondly, as dental restorative materials are exposed to foods and drinks of various compositions and pH, so, the effect of aging on VHN of the study groups should have been studied in multiple conditioning media like artificial saliva and some acidic media instead of only in deionized water to draw comparison of effects of aging on VHN by multiple conditioning media as well.

Conclusion

Long-term care of the materials surface quality is vital to improve the service life of esthetic dental restorations. RMGIC sustained its hardness upon prolonged aging in deionized water indicating its greater clinical tendency to maintain its structural dimensions as harder the dental restorative material is, less will it be susceptible to wear and vice versa. Hardness of GIC was low after day 1 but increased on day 912 which points out that greater care is needed for the GIC to gain sufficient strength / hardness especially after restoration placement during the first 1-2 days which can be ensured by advising the patient not to use that side for a couple of days. The hardness of Compomer and Giomer decreases on aging which indicates greater wear susceptibility of the dental restorations built with these materials. The decline in VHN values for Compomers is drastic when compared with Giomers owing to difference in setting reaction and filler loading.

Future Recommendations

There is a need to carry out similar studies to predict the wear potential of tooth-colored dental restorative materials using other parameters.

Authors Contribution

HG: Idea conception, study designing, data analysis, results and interpretation, manuscript writing and proof reading

MN: Idea conception, manuscript writing and proof reading

YJ: Data collection, manuscript writing and proof reading

MP: Data collection

TE: Data collection

UL: Data analysis, results and interpretation, manuscript writing and proof reading

SHH: Data analysis, results and interpretation, manuscript writing and proof reading

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