Spectrophotometric assessment of different surface coating materials on conventional glass ionomer cement

Aftab Ahmed Khan[1]*, Adel Zia Siddiqui[2], Jamaluddin Syed[3], Shafaq Noori[4] and Abdulaziz Abdullah Al kheraif[1]

[1]Dental Biomaterials Research Chair, College of Applied Medical Sciences, King Saud University, Riyadh, Saudi Arabia
[2]Dental Materials Department, Baqai Dental College, Baqai Medical University, Karachi, Pakistan
[3]Oral Basic and Clinical Sciences Department, Faculty of Dentistry, King Abdul Aziz University; Jeddah, Saudi Arabia
[4]Biochemistry Department, Muhammad Bin Qasim Medical and Dental College, Karachi, Pakistan

Abstract: The purpose of this *in vitro* study was to analyse the absorbance of dye material in conventional glass ionomer cement (GIC) by applying various commercially available surface protecting layers on GIC. 90 disc-shaped specimens were made using brass mold measuring 7mm in diameter and 2mm in thickness. 30 specimens were selected for each week testing having 6 groups (n=5). The groups were: G1 (Control group), G2 (Nail polish coated GIC), G3 (Master bond coated GIC), G4 (Copal varnish coated GIC), G5 (Varnal coated GIC), G6 (Cold mold seal coated GIC). The specimens of each group were immersed in a separate test tube filled with methylene blue dye, and placed in an incubator (37°C±2°C) for 1 week, 2 weeks and 3 weeks' time. After required time period, the specimens were rinsed under distal water for 1 minute and air dried for 1 hour. Next, the specimens of each group were put into new test tubes containing 1ml absolute alcohol and again stored at (37°C±2°C) for 24 hours. Absorbance were recorded in ultravoilet spectrophotometer. Results were analysed by Student *t*-test and Pearson's correlation. The results suggest that varnal and copal varnish are effective protecting materials with significant difference ($P<0.01$) after 3 weeks time. Our results conclude that the application of suitable protecting material may lead to longevity of GIC restorative biomaterial in a complexed oral environment.

Keywords: Spectrophotometer, Glass ionomer cement, Methylene blue, Surface protection.

INTRODUCTION

Fluoride releasing dental materials have been widely used due to their caries inhibiting properties (Hotwani *et al*., 2014). Glass ionomer cements (GICs) were invented by Wilson and Kent in 1972, set through an acid-base reaction occurring between polymers of polyacrylic acid and fluoroaluminosilicate glass (Berzins *et al*., 2010). GIC is famous for its slow release of fluoride ions, leading to its cariostatic action. It adheres chemically to enamel and dentin, biocompatible and serves in reducing the need for retentive cavity preparation (Ahluwalia *et al*., 2012). Moreover, GIC is a source of fluoride release which inhibits secondary caries (Kowsari *et al*., 2005). GIC also has the ability to recharge from topical applications containing fluoride. Different dispensing forms of fluoride including rinses, varnishes and gels are being used as topical fluoride applications which serve as "store house" for fluoride ions (Hedge *et al*., 2012). GICs are extensively used in preventing dental caries (Hotwani *et al*., 2013). The other important properties include biocompatibility with dental tooth structure, resistance to microleakage, acceptable marginal integrity and dimensional stability at high humid conditions and coefficient of thermal expansion near to tooth structure. However, there are some undesirable properties as well, such as, early moisture sensitivity, low wear resistance, poor strength and average aesthetics (Kamatham and Sharada, 2013).

Solubility and sorption is an important feature in evaluating the clinical longevity of dental cements. Therefore, solubility of dental cements has widely been considered for evaluation of both *in vitro* and *in vivo* studies. Water sorption and solubility may lead to degradation of cement, which may cause debonding of the restoration associated with recurrent decay (Ahmed, 2010). On the other hand, if GIC is exposed to air after initial setting, it will lose water immediately that may lead to shrinkage and crazing. This process will eventually leave the restoration surface prone to staining and place heavy loads on newly formed ionic bonds, which will result in loss of adhesion (Ribeiro *et al*., 1999). To reduce the susceptibility of conventional GIC, surface protecting materials have been proposed. In light of these suggestions, protecting agents referred to as coating agents or sealants have been specifically developed for rebonding of restorations (Karaoglanoglu *et al*., 2009).

Dental biomaterials undergo interaction with oral fluids with time. In few conditions the interactions may involve dissolution of surface properties whilst in others it may lead to leaching out of unbound or loosely bonded components, or uptake of fluids into the structure of the material (MaCabe and Rusby, 2003). The slow setting of

**Corresponding author:* e-mail: aakjk@hotmail.com

conventional GIC seems to be an inconvenient due to which GIC surface properties can easily be modified by dehydration or water uptake from saliva. If an immature dental restoration is not protected by varnish, the surface may deteriorate and result in dye infiltration (Davidson, 2006; Beriat and Nalbant, 2009). To overcome the problem of moisture sensitivity the use of different coatings such as water proof varnish, petroleum jelly, cocoa butter, or chemically/light cured bonding resins are applied (Hedge *et al*., 2012; Kamatham and Sharada, 2013). In the past different surface coating were recommended such as petroleum jelly, cocoa butter, waterproof nail varnish *etc*. With time, these coating deteriorate in harsh oral environment (Lohbauer, 2010; Cehreli *et al*., 2013).

Therefore, the aims of the present study is to compare the absorbance of methylene blue dye by conventional GIC using various commercially available surface protecting materials on the GIC. A spectrophotometer, which has the ability to quantitatively measure the reflection or transmission of a material (Anderson, 2004), was used to analyze the sorption rate. The null hypothesis is that protecting materials would not have any effect on the absorbance of the dye materials in GIC.

MATERIAL AND METHODS

Conventional GIC, Fuji II (GC Corporation, Tokyo, Japan) was chosen for this study. Using standardized brass mold, 90 disc shaped specimens 7±0.1mm in diameter and 2±0.1mm thick were fabricated at a room temperature (25ºC±2) and a relative humidity of 50%±10. The brass mold was pre-coated with a thin layer of petroleum jelly. GIC was mixed according to the manufacturer's instructions. To avoid any air bubbles, mold was covered with Mylar strip and compressed with glass slides from the upper and lower surfaces. Specimens were left for 10 minutes for setting. After setting, glass slides and mylar strips were removed. Discs with voids, bubbles and uneven rough surface texture were excluded from the study. For 1 week study 30 specimens were selected in which each group were having 5 specimens. Group 1: Control, group 2: Nail polish coated GIC, group 3: Master bond coated GIC, group 4: Copal varnish coated GIC, group 5: Varnal coated GIC and group 6: Cold mold seal coated GIC. Surface coating of these groups were performed with a painting brush and left for air dry. A single coat of these protecting materials were used on the representative specimens. The details of the protecting materials are presented in Table 1.

2% Methylene blue staining solution was prepared in laboratory by mixing 2ml of methylene blue dye material with 98ml of deionized water. Next, the specimens of each group were placed in a test tube filled with 1ml methylene blue dye and placed in an incubator (37°C±2°C) for 1 week. After 1 week, the specimens were rinsed under running distilled water for 1 minute and immersed into new test tubes containing 1ml absolute alcohol and again stored at (37°C±2°C) for 24 hours. The solutions were filtered and centrifuged (Centrifuge Model 800, China) for 3 minutes at 4000rpm and supernatant were used for analysis. Absorbance was recorded in Ultraviolet Vis Spectrophotometer (Schimadzu 160 UV-Vis, Germany) at 590nm and used for results. Same methodology was used in preparation of the specimens for 2 and 3 weeks study.

STATISTICAL ANALYSIS

Data were statistically analyzed using the SPSS 21.0 software program (SPSS®, Chicago, IL, USA). Student's *t*- test was used to determine if two sets of data are significantly different from each other. The degree of relationship between two variables was determined by Pearson's correlation. Significant differences between the control and the experimental groups were represented by $P<0.01$. Whereas, $P<0.001$ was considered highly significant.

RESULTS

Table 2 presents the mean values of the sorption rate at different point in time. The 7 days immersion of GIC specimens in methylene blue had a significant effect on the sorption rate of the GIC with cold mold seal. The control and other experimental groups showed adequate protection against the sorption rate ($P<0.001$, $P<0.01$). The GIC protected with copal varnish showed the least sorption rate among the tested groups in 7 days immersion in methylene blue dye ($P<0.01$).

2 weeks treatment of the GIC specimens in methylene blue dye showed detrimental effect on the sorption rate of the control, nail polish, master bond and cold mold seal groups. While the vernal and copal varnish groups ($P<0.0001$) showed no significant effect on the sorption rate ($P<0.0001$)

At the end of 21 days immersion of the specimens, the control, nail polish, master bond and cold mold seal groups showed diminished protection to the GIC surface ($P<0.001$, $P<0.01$). The copal varnish ($P<0.00001$) and vernal ($P<0.0001$) seemed to have adequately protected the GIC surface against the sorption of methylene blue dye. Figure 1 is showing a correlation curve between the week 1 and the week 3 treatment of methylene blue. The results indicate the strong negative correlation between the two treatments.

DISCUSSION

Oral fluids play a very important role in dissolution and disintegration of the restorative material. Effect of oral fluids has marked influence on the physical properties of

Table 1: Details of the protecting materials used in study groups

Surface Coating Agents	Composition
Nail Varnish	Camphor, Nitrocellulose, Sulfonamide, Toluene
Master Bond	Bisphenol A glicidylmethacrylate, Methacrylate groups, Ethyl alcohol, Sodium fluoride
Copal Varnish	Copal, Ethanol
Varnish (Varnal)	Resin staybilite (ester 10), Dimetylcetone
Cold Mould Seal	Sodium Alginate, Disodium Phosphate, Preservatives, Alcohol, Glycerine, Water

Table 2: Showing the mean and standard deviation of absorbance values and corresponding *P*-values of the tested groups

WEEK 1				
Test	Protective layers	n	Absorbance	*P*-value
T1	Control	5	0.0826±0.0028	0.002
T2	Nail Polish	5	0.072±0.002	0.002
T3	Master Bond	5	0.065±0.002	0.00001
T4	Copal Varnish	5	0.054±0.01	0.00046
T5	Varnal	5	0.062±0.0008	0.00001
T6	Cold mould seal	5	0.075±0.0054	0.038
WEEK 2				
Test	Protective layers	n	Absorbance	*P*-value
T1	Control	5	0.022±0.003	0.02
T2	Nail Polish	5	0.06±0.012	0.0019
T3	Master Bond	5	0.063±0.002	0.001
T4	Copal Varnish	5	0.25±0.009	0.0001
T5	Varnal	5	0.038±0.0032	0.0001
T6	Cold mould seal	5	0.11±0.008	0.1
WEEK 3				
Test	Protective layers	n	Absorbance	*P*-value
T1	Control	5	0.031±0.001	0.116
T2	Nail Polish	5	0.035±0.002	0.116
T3	Master Bond	5	0.027±0.026	0.632
T4	Copal Varnish	5	0.054±0.001	0.00001
T5	Varnal	5	0.042±0.002	0.0001
T6	Cold mould seal	5	0.038±0.002	0.31

GIC. High mechanical strength and low solubility of a restorative material is a desirable to bear the occlusal forces acting upon the restorative material (Kim *et al*., 2006).

In this study, the sorption rate of conventional GIC with different surface protecting materials were investigated. 2% methylene blue was used as a dye material in this study. Methylene blue is a common and easily available material used for spectrophotometric absorption. Being smaller molecular size, it has good penetration ability, because of this reason it is considered an ideal dye material (Modaresi *et al*., 2007). It was found that hydration properties of GIC without any surface protection and with cold mold seal protection was higher. Both GIC without any protection and GIC with cold mold seal protection found to have higher hydration properties when immersed in methylene blue dye for 1 week, 2 weeks and 3 weeks in comparison to other surface coating materials. Moreover, the sample of unprotected GIC and protected with cold mold seal were found to be fractured after 3 weeks period as a result of ingress of dye molecules. Therefore, we reject the null hypothesis.

The reason behind dissolution of the GIC in aqueous medium is could be the sodium presence, which forms a water soluble salt with the matrix, forming anions (Modaresi *et al*., 2007). Another theory explains that this could be attributed to washing of Ca^{+2} ions and Al^{+3} ions present in GIC that leads to weakening of acid base reaction (Kamatham and Sharada, 2013). These statistically significant dye uptake could be attributed to unprotected GIC and weakness shown by the cold mold seal as a protecting material. Among application of other surface protecting materials, no perceivable difference is found in the results, except that varnal and copal varnish

which showed adequate protection even after 3 weeks immersion in dye. To some extent master bond, which was able to protect the GIC specimen from significant water intake for two weeks and then lost the power to resist the dye penetration into the specimens.

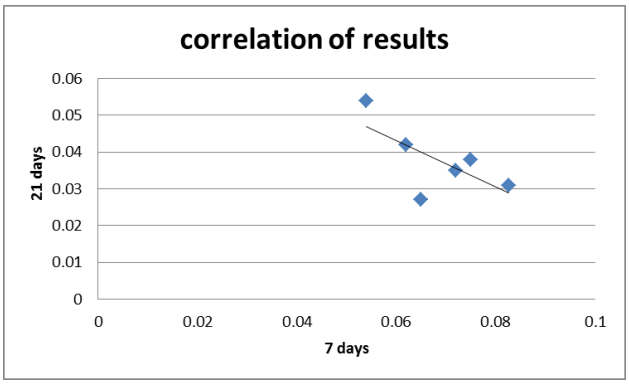

Fig. 1: Showing Pearson correlation curve for 7 days and week 3 study groups

In this study, varnal and copal varnish were the only coating materials found to be effective as compared to others. Result of this study is in accordance with the work done by (Cefaly *et al.*,2001; Serpil *et al.*, 2009; Fatima *et al.*, 2013; Brito *et al*., 2010) who all found nail varnish as an effective coating material for the longevity of a GIC in an oral environment. But their studies were not long enough to know the effect of dye penetration for upto three weeks. Our study also found the effectiveness of nail varnish. However, after week 2 the protective effectiveness started loosening (P=0.116).

Table 2 shows the dye penetration values for week 1, week 2 and week 3, respectively. It could be observed that all the surface protecting materials used on conventional GIC cement could not be able to completely block the imbibition of aqueous dye material. We can conclude that varnal, copal varnish and to some extent nail varnish were found to be protective materials in imbibition of the dye material. Other protecting materials were found to be sensitive against the dye intake.

The handling errors have always been an issue laboratory studies, Dye penetration test has some limitations as well. Acidic and alkalinity of a material may severely affect the dye penetration and could impair the result (Souza *et al*., 2009). One of the major limitations of this study is controlled *in vitro* environment in lieu of *in vivo* environment. No matter how ideal an *in vitro* environment be, it can not truly replicate the complex and dynamic oral environment.

CONCLUSION

Water movements across the set glass ionomer cement can be controlled by using effective surface protecting materials. We can conclude that the varnal and copal varnish protecting materials may significantly protect the surface and help in enhancing the clinical life of the glass ionomer cement.

ACKNOWLEDGEMENTS

The project was financially supported by Vice Deanship of Research Chairs, King Saud University, Saudi Arabia.

REFERENCES

Ahluwalia P, Chopra S and Thomas AM (2012). Strength characteristics and marginal sealing ability of chlorhexidine-modified glass ionomer cement: An in vitro study. *J. Indian Soc. Pedod. Prev. Dent*., **30**: 41-46.

Ahmed GM (2010). Water sorption and solubility of different commercially available dental cements (An in vitro study). *Med. J. Bab*., **7**: 410-421.

Anderson RJ, Bendell DJ and Groundwater PW (2004). Ultraviolet-Visible (UV-Vis) Spectroscopy. *In*: Abel EW editor. Organic Spectroscopic Analysis. Royal Society of Chemistry publishing, Cambridge, pp. 7-23.

Beriat NC and Nalbant D (2009). Water absorption and HEMA release of resin-modified glass-ionomers. *Eur. J. Dent*., **3**: 267-272.

Berzins DW, Abey S, Costache MC, Wilkie CA and Roberts HW (2010). Resin-modified Glass-ionomer Setting Reaction Competition. *J. Dent. Res*., **89**: 82-86.

Brito CR, Velasco LG, Bonini GA, Imparato JC and Raggio DP (2010). Glass ionomer cement hardness after different materials for surface protection. *J. Biomed. Mater. Res*., **93**: 243-246.

Cefaly DF, Seabra BG, Tapety CM, Taga EM, Valera F and Navarro MF (2001). Effectiveness of surface protection of resin modified glass ionomer cements evaluated spectrophotometrically. *Oper. Dent*., **26**: 401-405.

Cehreli SB, Tirali RE, Yalcinkaya Z and Cehreli ZC (2013). Microleakage of newly developed glass carbomer cement in primary teeth. *Eur. J. Dent.*, **7**: 15-21.

Davidson CL (2006). Advances in Glass-ionomer cements. *J. Appl Oral Sci*., **14**: 3-9.

Fatima N, Ali Abidi SY, Qazi FU and Jat SA (2013). Effectiveness of commonly available surface protecting agents in maintaining micro hardness of two cements. *J. Coll. Physicians Surg. Pak*., **23**: 315-318.

Hegde HM, Susan JJ, Darshana D and Hegde ND (2012). Effect of daily fluoride exposure on fluoride release by high strength glass ionomer restorative material used with atraumatic restorative technique: An *in vitro* study. *Int. Res. J. Pharm*., **3**: 241-246.

Hotwani K, Thosar N and Sudhindra B (2014). Comparative in vitro assessment of colour stability of hybrid esthetic restorative materials against various children's beverages. *J. Conserv. Dent*., **17**: 70-74.

Hotwani K, Thosar N, Baliga S, Bungale S and Sharma K (2013). Antibacterial effects of hybrid tooth colored

restorative materials against Streptococcus mutans: An *in vitro* analysis. *J. Conserv. Dent*., **16**: 319-322.

Kamatham R and Sharada RJ (2013). Surface coatings on glass ionomer restorations in pediatric dentistry-worthy or not. *J. Indian. Soc. Pedod. Prev. Dent*., **31**: 229-233.

Karaoglanoglu S, Akgul N, Ozdabak HN and Akgul HM (2009). Effectiveness of surface protection for glass-ionomer, resin-modified glass-ionomer and polyacid-modified composite resins. *Dent. Mater. J*., **28**: 96-101.

Kim TH, Jivraj SA and Donovan JTE (2006). Selection of Luting Agents: Part 2. *J. Calif. Dent. Assoc*., **34**: 161-66.

Kowsari A, Mahmoodian Jh and Ghavami T (2005). An in-vitro study on the release of fluoride from two restorative materials and their rechareability after exposure to daily 1000 ppm fluoride. *J. Dent*., **2**: 79-85.

Lohbauer U (2010). Dental glass ionomer cements as permanent filling materials?-properties, limitations and future trends. *Materials*, **3**: 76-96.

MaCabe JF and Rusby S (2003).Water absorption, dimensional change and radial pressure in resin matrix dental restorative materials. *Biomaterials*, **25**: 4001-4007.

Modaresi J, Davari AR and Daneshkazemi AR (2007). Comparison of apical leakage patterns shown by two different methods. *Pesq. Bras. Odontoped. Clin. Integr*., **7**: 169-172.

Ribeiro APG, Serra MC, Paulillo LAMS and Rodrigues AL (1999). Effectiveness of surface protection for resin-modified glass-ionomer materials. *Quintessence Int.*, **30**: 427-431.

Serpil K, Nilgun A, Hatice NO and Hayati MA (2009). Effectiveness of surface protection for glass ionomer, resin-modified glass ionomer and polyacid-modified composite resins. *Dent. Mater. J*., **28**: 96-101.

Souza EM, Pappen FG, Shemesh H, Bonanato-Estrela C and Bonetti-Filho I (2009). Reliability of assessing dye penetration along root canal fillings using methylene blue. *Aust. Endod. J*., **35**: 158-63