Evaluation of Solubility and Microleakage of Glass Carbomer Sealant

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Aim: This study was carried out to evaluate and compare solubility and microleakage of the newly introduced moisture tolerant glass carbomer sealant. Study design: For evaluation of solubility, 20 specimens of glass carbomer and conventional glass ionomer were prepared and immersed in artificial saliva of pH 4 and 6 for seven days. The difference between initial and final weight was calculated. For evaluation of microleakage, glass carbomer was compared with a conventional resin sealant. 20 premolar teeth indicated for orthodontic extraction were collected and divided into two groups and the respective sealants were applied. It was subjected to thermocycling and then kept immersed in methylene blue for 24 hours. Dye penetration was scored. Results: The glass carbomer specimens were less soluble than the conventional glass ionomer at both pH values. There was no significant difference in the microleakage. Conclusion: Being moisture resistant, glass carbomer can be used as an alternative fissure sealant material; especially in young children with partially erupted teeth and where obtaining moisture control is difficult.

Key words: solubility, marginal leakage, glass carbomer

INTRODUCTION

The anatomical pits and fissures of teeth which are generally considered as faults or imperfections in cuspal odontogenesis have long been recognized as susceptible areas for initiation of dental caries. The complex morphology of occlusal pits and fissures makes them an ideal site for retention of bacteria and food remnants, rendering the performance of oral hygiene difficult or even impossible¹. The extreme vulnerability to decay of these areas on the occlusal surfaces has prompted dental scientists to seek methods of caries prevention.²In the permanent dentition of young children, carious lesions most frequently develop in pits and fissures of first molars. Sealing those pits and fissures is considered the most effective way of interfering with the development of carious lesions over a long period.³

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Dr. Priya Subramaniam Department of Pedodontics and Preventive Dentistry The Oxford Dental College and Hospital Hosur Road, Bomanahalli, Bangalore 560068. India Phone: +91-80-30219733; 9844225624 Fax: +91-80-25734656 E-mail : drpriyapedo@yahoo.com A pit and fissure sealant is a resin based material that is introduced into the pits and fissures of caries-susceptible teeth, forming a micromechanically retained physically protective layer that acts to prevent demineralization of enamel by blocking the interactions of cariogenic bacteria and their nutrient substrate, thus eliminating the harmful acidic by-products.⁴ The properties required of an ideal fissure sealant include biocompatibility, anticariogenecity, adequate bond strength, good marginal integrity, insoluble in oral fluids, resistance to abrasion and wear and cost effectiveness.⁵

Resin and glass ionomer based materials have traditionally been used for sealing pits and fissures. An additional option for a sealant material with increased retention was introduced through the marketing of a novel glass-ionomer-based material – "the glass-carbomer", with powder particles reduced to nano size.⁵ Glass carbomer is a glass based material with an additional carbon chain and contains nano sized powder particles and fluorapatite as a secondary filler. The liquid of glass carbomer is polyacrylic acid.⁶ A major advantage of this material in pediatric dentistry is that it is moisture tolerant making it easy to place in children. Glass carbomer material is available for use as restorative material, sealants and for cementation of crowns and orthodontic bands.

Important factors in evaluation of sealant success are marginal integrity and solubility. Marginal integrity can be appreciated by evaluating microleakage, which is defined as the ingress of oral fluids into the space between the tooth and restorative material. In vitro microleakage studies make it possible to predict the marginal sealing ability of materials. ⁷ Solubility of the material may cause degradation of the cement, leading to debonding of the sealant and a break in the marginal integrity and cause recurrent decay. ⁸

As glass carbomer is a newly introduced material which has not yet been approved for clinical use, not many studies have been done evaluating its physical properties. Hence this study was carried out to evaluate and compare solubility and microleakage of glass carbomer sealant.

MATERIALS AND METHOD

Evaluation Of Solubility

The glass carbomer sealant material (GCP Dental, The Netherlands) and the conventional glass ionomer, indicated for sealant application (Fuji type VII (GC Corporation. Tokyo, Japan) were selected for this study. For the standardization of specimens, a custom made cylindrical plastic mould of dimensions: 6 x 4 x 10mm length was used to prepare the specimens.

The glass carbomer sealant material is supplied as capsules. Prior to mixing, the capsules were inserted into a universal capsule gun and standardized according to manufacturer's instructions. It was mixed for 7 seconds in a high frequency amalgamator. The pin from the nozzle was removed after mixing and it was inserted into the capsule gun and the lever was pulled twice to prime the material which was then extruded onto the custom made mould. It was pressed down using a Mylar strip to remove the excess material. A layer of GCP Gloss (GCP Dental, The Netherlands) was applied on the surface of the material. It was then cured using a LED curing unit (GCP Carboled CL 01) with an output of 1400 mw/cm² for 60 seconds. A total of twenty specimens of the glass carbomer sealant material were prepared (group 1).

For the preparation of samples of conventional glass ionomer material, the powder and liquid of Fuji type VII GIC were mixed according to the manufacturer's instructions in a powder/liquid ratio of 1:1. Two scoops of powder was mixed with two drops of liquid on an impermeable paper using an agate spatula for thirty seconds. The mixed material was immediately placed in the mould, which was coated which a thin layer of petroleum jelly to prevent material adhesion. The mould was slightly overfilled and was then pressed on top with a Mylar strip to remove the excess material and to prevent air bubble formation. A total of twenty specimens were prepared (group 2).

After setting, the specimens of both the materials were removed from the mold and the excess was trimmed using a Bard Parker blade.

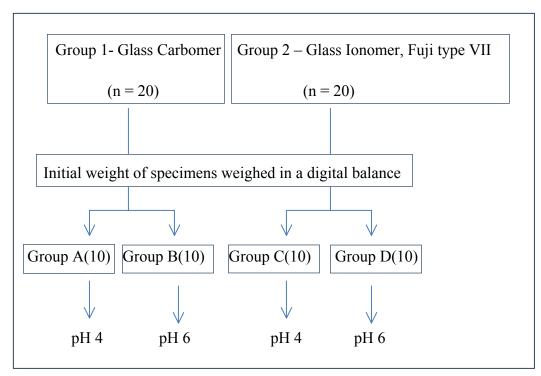
Experiment procedure

Initial weights of the forty specimens were weighed individually using a precision digital balance (Mettler A J150). The specimens of group 1 were then further subdivided into groups A and B, each consisting of ten specimens. Similarly group 2 was subdivided into groups C and D, consisting of ten specimens each.

Artificial saliva was prepared according to the composition given by Fusayama *et al*⁹. Ten specimens from group 1 and group 2 (group A and C) were immersed in the artificial saliva at pH 4 and the other ten specimens (group B and D) into artificial saliva at pH 6.⁹ (Table 1). They were stored for 7 days at $37\pm1^{\circ}$ C.

On each day, the specimens were removed from the artificial saliva with a pair of tweezers, blotted with clean absorbent paper until their surfaces became free from visible moisture and waved in air for 15 seconds. It was then stored in a desiccator containing anhydrous calcium sulfate for 60 minutes. The specimens were then

Table 1: Procedure protocol for evaluation solubility of Glass Carbomer sealant material and Conventional glass ionomer i.e Fuji Type VII material in Fusayama artificial saliva of pH values of 4 and 6.



weighed using the precision digital balance. Amount of weight loss was calculated as the difference between the initial weight of the specimen and its final constant weight after drying in the desiccator.

Evaluation of Microleakage

The two materials used for the evaluation of microleakage were glass carbomer sealant (GCP Dental, The Netherlands) and the traditionally used conventional resin sealant (Embrace WetBond, Pulpdent Corporation, MA, USA) which were commercially obtained.

Twenty freshly extracted, sound premolar teeth, indicated for orthodontic purposes, were collected and stored in 0.1% thymol. The teeth were free of restorations, fluorosis, caries and sealants. The occlusal surface of the teeth were thoroughly cleaned with hand scaling instruments to clear them of debris and followed with a slurry of pumice. The teeth were then divided into two groups of ten teeth each (group 1 and group 2).

For the teeth in group 1, glass carbomer sealant material was applied. The capsule of glass carbomer was activated as described earlier. The material was extruded on to the occlusal surface of the tooth and spread as a thin film ensuring that no air bubbles were included. With the help of a cotton pellet, a thin layer of GCP gloss (GCP Dental, The Netherlands) was applied over the sealant. It was then cured using a LED curing unit (GCP Carboled CL 01) with an output of 1400 mw/cm² for 60 seconds.

For the teeth in group 2, the conventional resin sealant was applied. The occlusal surfaces were etched with 37% phosphoric acid (Etch-Rite, Pulpdent Corporation, MA, USA) for 15 seconds and rinsed with water. With Embrace WetBond (Pulpdent Corporation, MA, USA), the typical dull, frosted appearance of the etched surface is not desired, rather, the surface should be lightly dried and slightly moist with a glossy appearance. The sealant Embrace wet bond was applied as per manufacturer's instructions and a probe was used to flow the sealant and prevent air bubbles. It was then light cured for 20 seconds.

The treated teeth of both groups were stored in sealed containers containing distilled water, in a laboratory oven at 37°C for 24 hours. The teeth were then subjected to thermocycling for 200 cycles between 5 and 55°C with a dwell time of 15 seconds and a transfer time of 15 seconds to simulate the temperature changes that occur in the oral cavity. All tooth surfaces with the exception of a 1 x 1 mm window around the sealant margin was double coated with finger nail varnish. The teeth were then immersed in 5% methylene blue for 24 hours. After removal from the dye, they were rinsed with distilled water to remove superficial dye. A diamond disc at slow speed was used to section the teeth longitudinally in a bucco-lingual direction. The sectioned teeth were examined under the stereomicroscope at 35X magnification and scored according to criteria given by Chaitra *et al.*¹⁰

- 0 No dye penetration.
- 1 Dye penetration up to 1/3 of the depth of the fissure.
- 2 Dye penetration more than 1/3 and less than 2/3 of the depth of the fissure.
- 3 Dye penetration more than 2/3 of the depth of the fissure.

Data obtained for both parameters was subjected to statistical analysis. Group wise comparison was made using Mann Whitney

U test. Intra group comparison was made using Wilcoxon matched pairs test. A p value of < 0.05 was considered as statistically significant. Analysis was carried out using the SPSS- Version 16 software.

RESULTS

Solubility: At pH 4 and 6, glass carbomer sealant showed a significant increase in weight. At pH 4, the increase in weight was from day two and, at pH 6 it was from day five, which remained steady till day seven. The conventional glass ionomer sealant did not change weight till day six after which it showed a decrease in weight on day seven. (Graph 1 and 2). Inter group comparison showed that there was a significant difference in weight between the two materials only on day seven, at both pH values.

Microleakage: The mean microleakage score for group 1 was 2.4 ± 1.07 and for group 2 it was 1.7 ± 1.07 . (Table 2). There was no significant difference between the two materials. However, glass carbomer showed majority of the specimens had dye penetration beyond 2/3rd the of the fissure depth, whereas, in the conventional resin sealant majority of the specimens had dye penetration to only 1/3rd of the fissure.

 Table 2: Comparison of mean microleakage scores between the test groups

Study groups	Mean ± SD	p-value
Group 1	2.4±1.07	
Group 2	1.6±1.07	p=0.151

DISCUSSION

Glass carbomer is a relatively new material which is a modification of glass ionomers. It is used as a fissure sealant that is designed to deliberately remineralize in the mouth. Glass carbomer is claimed to contain nanocrystals of calcium fluorapatite, which can act as nuclei for the remineralization process and initiate the formation of fluorapatite. It has a much finer particle size compared to conventional glass ionomer cements. 11,12 The nano-sized particles would facilitate a strengthening of the material through an increased particle surface in contact with the glass-carbomer liquid and would aid its dissolution and ultimate conversion to fluorapatite.^{13,14} This would increase the compressive strength and wear resistance of the material.⁶ Addition of hydroxyapatite and fluorapatite are also reported to enhance the mechanical properties of the material.^{11,15} The reactive glass is treated with dialkyl siloxanes described in the European Patent 20040748628.6 According to the manufacturer, glass carbomer sets chemically and is optimized for heat curing. An advantage of glass carbomer is that it is moisture tolerant and is easy to use in children in whom moisture control can be a challenge. However, not many studies have been done evaluating the physical properties of the glass carbomer sealant and hence this study was taken up to evaluate solubility and microleakage of the material.

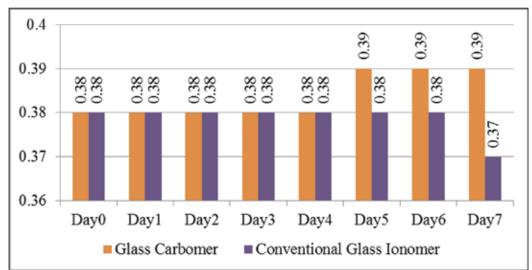
Solubility or leaching of cement components has a potential impact on both its structural stability and biocompatibility. The rate of dissolution can be influenced by the conditions of the test. Other factors may include time of dissolution, concentration of solute in the dissolution medium, pH of medium, specimen shape and thickness, and powder/liquid ratio of cement.¹⁶

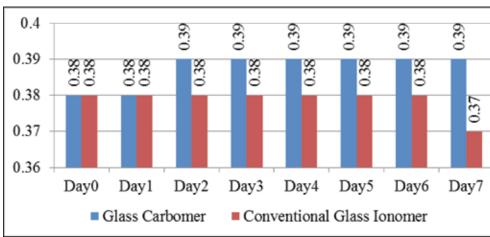
The method used for testing the solubility in this study followed a modification of section 7.12 of ISO 4049. The test requires that specimens be first placed in a desiccator immediately after curing and removal from the mold. In this study, section 7.12 was modified by placing specimens in a solution of artificial saliva immediately after preparation.¹⁷ The rationale behind this modification was to ensure that no desiccation of glass ionomer specimens occurred immediately after fabrication as it might affect their solubility and sorption results due to damage. For purpose of standardization, all specimens were immediately placed in artificial saliva after removal from mould.¹⁸

The chemical structure of solutions used for in vitro tests is important because it has to simulate the complexity of the oral environment. The in vitro tests made are only static solubility tests because they do not simulate the pH and temperature changes of the oral cavity.¹⁹ In studies pertaining to dental cements; solvents such as water and acids have been used to act as food-simulating liquids. ^{20,21} Levine et al, suggested the use of artificial saliva to produce a setting similar to the oral medium. ²² Lower pH values increase the solubility of cements. ^{23,24} Walls et al, obtained highest solubility at pH 4, whereas no dissolution was observed in a buffer solution at pH 10 even after 24 hours for glass ionomer cements. ²⁵ Therefore, artificial saliva of pH values at 4 and 6 were prepared for this study.

Glass carbomer material showed an increase in weight at both pH of 4 and 6 in contrast to glass ionomers, which showed a decrease in weight on day 7. This increase in weight of glass carbomer may be because the basic constituents of glass ionomer cement, polycarboxylic acids and ion leachable glass, bind water molecules. Since the composition of glass carbomer is 100% glass, an increased concentration of glass may lead to water absorption and hence increased weight. This could lead to poorer clinical performance as usually a serious warpage and dimensional change in the material are associated with water absorption.²⁶ As the material was stored in artificial saliva, weight increase may have also been due to uptake of calcium and phosphate released by the artificial saliva. On communication with the manufacturers of glass carbomer, it was explained that as it is a biomimetic material, the increase in weight may also be due to the formation of fluorapatite crystals. However, a study done on the characterization of remineralizing ability of Glass Carbomer by MAS-NMR Spectroscopy revealed that the apatite in this cement was not fluorapatite but largely hydroxyapatite.13 Therefore, further studies need to be carried out to determine the nature of remineralization of glass carbomer.

Graph 1 - Mean weight of specimens at pH 6





Graph 2 - Mean weight of specimens at pH 4

An important concern in restorative dentistry is microleakage occurring at the tooth-restoration interface. It is the clinically undetectable passage of bacteria, fluids, molecules or ions between the cavity wall and the applied restorative material. With pit and fissure sealants, microleakage can result in initiation and/or progression of caries under sealed surfaces; as well as increasing the difficulty of diagnosing and treating this lesion. Consequently, microleakage can be considered the main detrimental factor that affects the sealant efficiency. ¹⁰Resin sealants are most commonly used as pit and fissure sealants in clinical practice and hence it was used for comparison with glass carbomer. Further research is proposed in our department, to compare glass carbomer and glass ionomer in an in-vivo condition and hence a comparison with glass ionomer was not included for the in-vitro study.

In the present study, glass carbomer group showed higher values of dye penetration compared to the conventional resin sealant. Majority of the specimens of glass carbomer showed dye penetration beyond 2/3 of the fissure depth, whereas with the conventional resin sealant dye penetration was up to 1/3 of fissure depth. An earlier study on the microleakage of glass carbomer using micro CT reported of crack patterns, referred to as "fracture lines" and the results were non-interpretable. ²⁷ Thus, in our study we used the dye leakage method to evaluate microleakage of glass carbomer.

Glass carbomer material is more viscous than conventional resin material. A low viscous sealant has a greater potential to flow, spread more rapidly over the surface and penetrate into the pits and fissures.²⁸ This could be a reason why there was incomplete penetration of glass carbomer into the depth of fissures, leading to higher scores for dye penetration.

In the present study, distilled water was used to store the specimens for 24 hours at 37 C and this may have influenced the results as distilled water leads to ion loss as it acts as a magnet for ions, hence leading to increased microleakage scores in both groups. Pooled human saliva was not used in our study as it could lead to microbial contamination of the specimens.

A protective surface coating is recommended by the manufacturers for application over glass carbomer sealant. The GCP gloss (GCP Dental, The Netherlands) is a silicone-based coat to protect the surface from exposure to moisture and saliva during the first setting reaction and from dehydration in the second phase.²⁹ A previous study concluded that greatest amount of microleakage was observed in uncoated glass carbomer specimens as compared to specimens with surface coating.⁶ In our study, a thin layer of surface coating was applied on the specimen. This could be the reason for observation of microleakage that was comparable to that of conventional resin sealant.

Glass carbomer is an esthetic material which over a period of time resembles the natural tooth colour. This is due to the occurrence of natural mineralization which gradually increases the transparency to match with the surrounding dentition.

Future investigations on surface topography of the material need to be done using Scanning Electron Microscopy (SEM) and Surface photomicrography.

Being moisture resistant, glass carbomer can be used as an alternative fissure sealant material; especially in young children with partially erupted teeth and where obtaining moisture control is difficult. However, investigations on the clinical performance of this material are required before it can be recommended as an alternative for glass ionomers.

CONCLUSION

- 1. The solubility of glass carbomer sealant was lesser than the conventional glass ionomer sealant at both pH of 4 and 6.
- 2. There was no significant difference in microleakage between glass carbomer sealant and the conventional resin sealant.

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