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Properties of Glass-Ionomer Cements Sealed with Petroleum Jelly or Wax

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Abstract**—**A study has been carried out to determine the effect of coating two commercial glass-ionomer cements in either petroleum jelly or wax. After coating, specimens were stored in water for 24 or 168 hours, then the coating removed and the surface examined. Coating in wax was found to increase the surface hardness significantly compared with the uncoated control, whereas coating the specimens in petroleum jelly led to only a slight increase in surface hardness. Coating in wax led to no detectable ion release after either 24 or 168 hours, though there was some ion release after the coating had been removed and the specimens exposed to water for a further 24 hours. This shows that soluble species remained in these specimens. Overall, this study confirms the idea that immature glass-ionomers should be protected from early exposure to moisture, and that the protection offered by petroleum jelly is only modest.

Keywords**—**Coating, Glass-Ionomer Cements, Ion Release, Surface Hardness, Wax.

I. Introduction

GLASS-IONOMER CEMENTS (GIC) were introduced to clinical dentistry in the late 1970s as a result of the pioneering work of Wilson and Kent [1], [2]. These early GICs had very inferior properties compared to the materials available today [3], though their essential components and their setting chemistry, *i.e.* reaction of an acid with a basic glass powder, have remained the same.

The setting reaction of a GIC continues slowly after the initial hardening is complete, and may go on for more than twenty-four hours. During this time, the water within the restorations is still labile, and may be lost on exposure to dry air. This results in the development of microcracks in the surface and a chalky appearance. Restorations may also be susceptible to attack by saliva, with consequent washing out of matrix-forming ions. This results in loss of translucency, dimensional changes and reduction in physical strength [4], [5]. Glass-ionomers are particularly sensitive to this in the first hour after placement [4].

To maintain the necessary water content of GICs and to protect them from premature exposure to moisture for the first few hours after placement, several methods are available to the clinician. These include solvent-based resins and varnishes, light-curable varnishes [6], and emollients such as petroleum jelly [7], [8].

Fluoride release is an important property of glass-ionomers [8]. Studies have shown that varnishes reduce fluoride release by between 61 and 76%, depending on the cement and the varnish [9]. Thus varnishes have a negative effect on one of the desirable features of these cements.

Varnishes affect two features of water loss, namely rate and end point [7], [11]. Water loss from uncoated glass-ionomers has been shown to be a diffusion process [1], with a simple linear relationship between mass loss and square root of time. The presence of varnishes alters this, so that water loss is no longer a simple process, and does not follow a straightforward linear relationship with √(time) [11]. In addition to altering the mechanism of water loss, varnishes generally reduce the amount of water lost at equilibrium [7].

As well as specially formulated varnishes, glass-ionomers can be protected from early dehydration and/or exposure to water by coating with petroleum jelly [3]. So far, little information has appeared in the literature of the effect of this treatment on the properties of glass-ionomers. Also, there is no information on the properties of glass-ionomers that have been completely protected from exposure to the surroundings. This is possible by coating them in wax, and leaving the coating in place for varying lengths of time. The current study addresses both of these topics. The aims of this in vitro study were to 1) examine the effects of petroleum jelly and wax on the Vickers Hardness of glass-ionomers, 2) to analyse the differences in ion release from the materials coated in these ways; and 3) to examine the surface morphology of the specimens once the coatings had been removed.

II. Materials and Methods

Standard specimens were prepared from two conventional glass-ionomer products; Fuji IX (GC, Tokyo, Japan) and AquaCem (DENTSPLY, UK). Fuji IX is based on a strontium-containing glass whereas AquaCem is based on a calcium glass. Two different cement compositions were chosen to establish if the effects of the coatings were element-specific. The powder and liquid of both products were weighed, and the cements hand mixed according to the manufacturers' instructions. Powder:liquid ratios were, respectively, 3.6:1 and 5.0:1 for Fuji IX and AquaCem, with the liquid being either aqueous polyacrylic acid, as supplied, or deionised water. Freshly mixed pastes were placed in

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silicone rubber moulds and pressed between two glass slides to eliminate excess material and air bubbles. This technique also prevents early dehydration of the cement. Disc-shaped specimens of 4 mm diameter and 2 mm height were made. The cements were left in an oven at 37 °C for 1 h to cure fully.

After curing, the cements were removed from their moulds and divided into Group 1: Control, no surface protection, immersed immediately in 10 ml distilled water; Group 2: Coated with petroleum jelly (P.J.), BP grade, and immersed in 10 ml distilled water; and Group 3: Coated with paraffin wax, then immersed in 10 ml distilled water. Samples were stored in their designated conditions for either 24 h or 1 week (168 hours) at 37 °C, after which time the coatings were removed from the samples and the surface hardness was tested. All studies were performed in triplicate (Three samples per group).

The Vickers Hardness measurements were performed on the Buehler high quality hardness tester #1600-1625 using a diamond indenter with 200 g of load and 20 s dwell time. For each specimen, three random measurements were made on either side of the disc and the mean determined, making a total of six measurements for each specimen. Student's t-test was used to analyse the results for significant differences between the coated and control specimens.

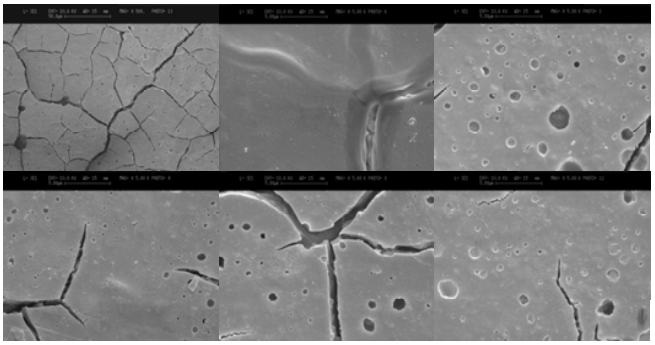
The surface of a representative specimen for each group was later analysed for changes in surface morphology using a Cambridge Stereoscan 360 SEM. All images were taken at a magnification of X5000 so that a direct comparison could be made.

A duplicate experiment with identical conditions was set up to determine ion release. After the designated time periods the samples were removed from the distilled water, which was stored for analysis, the surface coatings were removed and finally samples were placed in a fresh 10 ml of distilled water for a further 24 h. After this time, both sets of storage liquors were analysed using ICP-OES (Perkin-Elmer Optima 4300DV).

III. Results

Fig. 1 shows SEM images (a–f.). Images a–c show Fuji IX specimens stored for 24 h with no coating, petroleum jelly and wax respectively. Images d–f show Fuji IX specimens stored for 168 h with no coating, petroleum jelly and wax respectively.

a) Fuji IX 24 h water b) Fuji IX 24 h P.J. c) Fuji IX 24 h wax

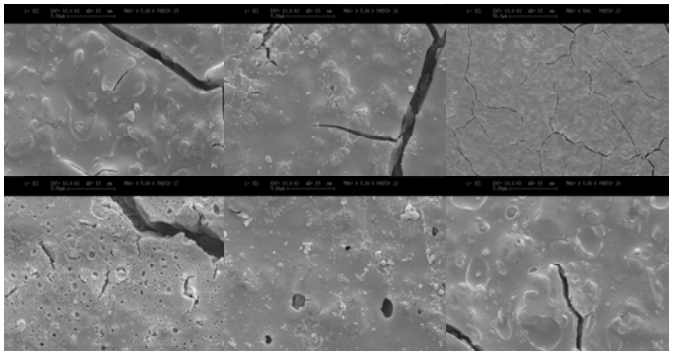


d) Fuji IX 168 h water e) Fuji IX 168 h P.J. f) Fuji IX 168 h wax

Fig. 1: SEM images of Fuji IX stored for 24 and 168 hours in different coatings

Fig. 2 shows the equivalent SEM images for AquaCem specimens. It can be seen from the images that the various treatments do not appear to have altered the surface morphology of the cement. The cracks are caused by the high vacuum inside the SEM which dehydrates the material and the small 'craters' on the surface are the result of air bubbles being present in the cement due to hand mixing.

a) A.C. 24 h water b) A.C. 24 h P.J. c) A.C. 24 h wax



d) A.C. 168 h water e) A.C. 168 h P.J. f) A.C. 168 h wax

Fig. 2 SEM images of AquaCem stored for 24 and 168 hours in different coatings

Table I shows the mean and standard deviation hardness values for Fuji IX and AquaCem after being coated and stored for 24 h and 1 week. The values for AquaCem are lower than those of Fuji IX due to the high water content in the cement. For both cements at both storage times, specimens coated in wax had the highest mean hardness values whereas the uncoated control had the lowest. No significant differences were found between the petroleum jelly and water stored samples independent of the cement composition. However, the differences between the wax coated samples and the control were significant ($p<0.001$) for both cement types.

TABLE I
VICKERS HARDNESS NUMBERS FOR CEMENTS STORED IN DIFFERENT COATING MATERIALS FOR 24 AND 168 HOURS (STANDARD DEVIATIONS IN PARENTHESES)

Cement Type	Surface Coating Material	Vickers Hardness 24 h	Vickers Hardness 168 h
Fuji IX	Control (water)	71.9 (2.8)	75.2 (3.2)
	Petroleum Jelly	74.4 (1.8)	79.7 (4.5)
	Wax	181.6 (4.6)	112.3 (2.2)
AquaCem	Control (water)	35.5 (0.7)	34.8 (0.5)
	Petroleum Jelly	38.4 (1.3)	40.8 (0.2)
	Wax	74.9 (1.6)	72.7 (2.3)

Table II shows the ICP-OES results for Fuji IX before and after the coatings were removed and Table III shows the ICP-OES results for AquaCem before and after the coatings were removed. It can be observed from the tables that petroleum jelly coating reduces the release of all ions with the exception of Sr in Fuji IX compared to the control. Wax has shown to prevent the release of all tested ions from both AquaCem and Fuji IX. After the coating was removed and the samples re-submerged in water the ion release was shown to be lower in all cases with the exception of Ca from AquaCem.

TABLE II
ION RELEASE INTO WATER FROM FUJI IX SPECIMENS STORED IN DIFFERENT COATINGS (STANDARD DEVIATIONS IN PARENTHESES)

Time In Coating	Ion Species	With coating			After coating was removed		
		Control	Petroleum Jelly	Wax	Control	Petroleum Jelly	Wax
24 h	Sr	0.05 (<0.01)	0.12 (0.02)	b.d.l.	0.01 (<0.01)	0.02 (<0.01)	0.01 (<0.01)
	Al	0.25 (0.08)	0.07 (0.05)	b.d.l.	<0.01 (<0.01)	0.10 (0.04)	0.10 (0.04)
	Si	0.78 (0.11)	0.23 (0.10)	b.d.l.	0.31 (0.09)	0.25 (0.05)	0.22 (0.03)
168 h	Sr	0.18 (0.02)	0.06 (0.02)	b.d.l.	0.02 (0.01)	0.02 (0.01)	0.02 (<0.01)
	Al	0.27 (0.02)	0.12 (0.06)	b.d.l.	<0.01 (<0.01)	<0.01 (<0.01)	0.09 (<0.01)
	Si	2.05 (0.12)	0.47 (0.08)	b.d.l.	0.06 (0.01)	0.01 (<0.01)	0.24 (0.04)

b.d.l. = below detection limit

TABLE III
ION RELEASE INTO WATER FROM AQUACEM SPECIMENS STORED IN DIFFERENT COATINGS (STANDARD DEVIATIONS IN PARENTHESES)

Time In Coating	Ion Species	With coating			After coating was removed		
		Control	Petroleum Jelly	Wax	Control	Petroleum Jelly	Wax
24 h	Ca	0.03 (0.01)	0.01 (<0.01)	b.d.l.	0.06 (0.01)	<0.01 (<0.01)	0.60 (0.03)
	Al	4.27 (0.36)	2.96 (0.55)	b.d.l.	1.46 (0.31)	1.08 (0.22)	1.72 (0.19)
	Si	14.77 (1.85)	10.02 (0.78)	b.d.l.	8.24 (0.88)	6.73 (0.84)	5.57 (0.35)
168 h	Ca	2.81 (0.10)	2.19 (0.40)	b.d.l.	1.25 (0.13)	0.83 (0.16)	0.70 (0.28)
	Al	7.96 (0.39)	6.41 (0.89)	b.d.l.	0.66 (0.02)	0.50 (0.10)	1.14 (0.39)
	Si	39.61 (1.28)	33.36 (4.37)	b.d.l.	5.33 (0.09)	4.48 (0.47)	3.81 (0.61)

b.d.l. = below detection limit

IV. DISCUSSION

The setting reaction of a GIC can be affected by contact with water in several of its setting stages [13], [14]. The reaction starts when an aqueous solution of polycarboxylic acid is mixed with glass particles rich in calcium or strontium, aluminium and silica. As the powder and liquid combine, cations from the glass are leached into the aqueous acidic phase, where they interact with the carboxylate groups to form a cross-linked polyacrylate chain matrix. As this progresses, the material hardens from a paste into a solid mass. This process normally takes 2-10 minutes [1]. Water contact at this stage of the reaction can cause irreparable damage to the cement, as cross-linking ions are washed out of the matrix. However, as the setting reaction continues, aluminium is

incorporated into the polyacrylate matrix and the cement becomes less vulnerable to damage by ion wash-out. During this early stage of reaction, the cement is also sensitive to loss of water, and will dehydrate when exposed to air [12], [15]. This also leads to damage, with the formation of numerous surface micro-cracks and the development of a chalky appearance. To protect the cement from both of these potentially adverse effects, in clinical use, the cement is coated with a waterproof layer, either of petroleum jelly or of varnish, until this vulnerable stage has passed.

Coating cement samples in wax was found to lead to the greatest levels of surface hardness, with values significantly higher ($p<0.001$) than those stored in petroleum jelly or water.

This confirms that early exposure of an immature glass-ionomer cement to water leads to damage [16]. Although the specimens coated in petroleum jelly were slightly harder than those exposed to water, the effect was slight, particularly compared to the effect of coating in wax, showing that coating with this substance provides only modest protection against the adverse effects of exposure to water.

Ion release values confirmed these inferences. Those for the control were the highest, showing that more ions were dissolved from the immature cement than from those coated with either petroleum jelly or wax. In the case of wax, ion release for all species examined was below the limit of detection, confirming that wax gave the best protection from attack by water. Patterns of ion release following removal of the coatings were variable, and in certain cases, for example for aluminium released from AquaCem, were greater in the specimens that had been coated in wax. This shows that not all of the ionic species become completely insolubilised after 168 hours, but that the wax coating had been able to retain them in the cement. This retention appears to be desirable in promoting the highest possible surface hardness.

The SEM images of the surfaces show that the various treatments have only minor, if any, effects on the surface morphology. All of the samples show essentially the same features, namely surfaces with unreacted glass particles embedded in a polyalkenoate matrix. There are no major morphological differences, suggesting that there is no direct link between appearance and surface hardness in these materials.

In terms of clinical significance, the paraffin wax used in this study could be applied in the oral cavity but further studies would need to be carried out to determine if any detrimental effects occur.

V. Conclusion

This study has shown that coating glass-ionomer cements in wax shortly after setting, significantly increases the surface hardness of the material that develops at 24 and 168 hours. By contrast, storing the specimens in petroleum jelly leads to only a slight increase in surface hardness. The values obtained when storing specimens in wax are much higher than any previously reported for these materials, showing that there is considerable scope for improving their surface hardness values, given the correct conditions.

The detrimental effect of water has been confirmed, since exposure of uncoated cements to water led to low surface hardness values and relatively high levels of ion leaching. Specimens stored in petroleum jelly showed some improvement in surface hardness and reduced ion leaching compared with those stored in water, however the effects were slight and considerable less than when coating specimens with wax. It is clear from the current study that petroleum jelly is only moderately effective at protecting the cement from water and that the cement matures to a state of increased surface hardness if cured in an environment completely protected from exposure to water. This confirms that the current clinical practise of protecting newly placed glass-ionomer cements from early exposure to moisture is appropriate, but that petroleum jelly offers only slight protection.

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