DO GLASS IONOMER CEMENTS MATURE APPRECIABLY? A CRITICAL REVIEW

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Abstract

Introduction: The earliest glass ionomer cement (GIC) had low strength when tested after 24 hours. However, after long periods, strength increased. This was initially thought to be a property of all GICs, but some later materials either did not increase or got weaker. Objectives: This review aims at determining whether changes in the chemical composition of the glass component of GICs do result in different strength changes or whether other formulation changes are also responsible. In addition, it aims at evaluating changes in other non-mechanical properties, such as abrasion and erosion. **Materials and methods:** Search engines were used to find relevant references included in Ovid SP, Pubmed and Index Copernicus.**Results and discussion:** In experimental GICs strength, changes have an inverse correlation with initial (24 hours) strength. For commercial GICs, no such relationship is found. High phosphorus contents result in large increases in strength from initial low strength values. Other properties also show maturation changes. In particular, both resistance to abrasion and erosion increase in the relatively few GICs tested, including some which do not show strength increases. **Conclusions:** GICs have the potential to improve their properties when matured for periods longer than 24 hours, however this property is dependent on glass composition.

Keywords: *glass ionomers, compressive strength, maturation*.

1. INTRODUCTION

The first commercial dental glass ionomer cement (GIC) Aspa inherited two clinically important characteristics from its two "parents". From zinc polycarboxylate cement (ZPC) came the direct adhesion to hard tooth tissues from the shared polymeric acid component. From the glass component shared with its dental silicate cement (DSC) "parent" came the prolonged release of fluoride ions.

The mechanical properties of Aspa were not comparable with those of DSCs, used for 60 years as permanent fillings. This was indicated by the requirements of the test standards for compressive strength, dropped from 169MPa for DSC [ISO1565] to 125MPa for GIC [ISO7489]. This test, in common with most dental standard strength tests, is carried out after 24 hours. It was reported that at times longer than 24 hours, the strength of Aspa increased [1]. The authors commented that: "The strength of 1-year specimens was considerably greater than that of 24-hour old specimens." The increase was 35%, most of it (26.5%) occurring in the first month. All test specimens were stored in water at 37ºC, as specified by the test standards. However, if stored in a "non-volatile paraffin liquid", the increase within one year was ~80%. Tests on subsequent commercial GICs (stored in water) indicated that, whereas some also showed enhanced compressive strength beyond 24 hours, others showed no change and some showed an appreciable decrease [2].

The literature indicates that maturation produces important changes in cement properties. In addition to strength properties (flexural as well as compressive), changes have been reported in relation with erosion, abrasion, hardness, adhesion, creep/ stress relaxation, fracture toughness, modulus and translucency/ colour.

The aim of this review is to examine the reported maturation changes in GIC properties and the mechanisms proposed to explain them.

2. MATERIALS AND METHODS

Search engines were used to find relevant references included Ovid SP, Pubmed, and Index Copernicus. Keywords used were "Glass Ionomer

Cement", "Glass Polyalkenoate Cement", "Maturation", and "Mechanical Properties". Original research publications published in English were used in this study. Review articles, those not peer-reviewed and not elaborated in English were excluded.

3. RESULTS AND DISCUSSION

COMPRESSIVE STRENGTH

Figures for 23 commercial materials in addition to Aspa are available. The figures recorded at 24 hours and 28 days are available in each case, being listed in Table 1.

Table 1. Compressive strength change commercial restorative GICs

Details	CS 24hr MPa	CS 28day Mpa	Δ CS ± %	Sig Level
Aspa	170	215	+26.5	N/A
ChemFil II	207	183	-11.6	N/A
Opusfil W	225	250	+11.1	N/A
RGI Core Build	265	264	-0.4	N/A
Fuji II (R)	209	210	+0.3	N/A
Diamond	215	234	+8.8	N/A
HiFi	234	226	-3.4	N/A
HiDense	244	291	+19.3	N/A
Chelon	251	228	-9.2	N/A
Ketac	247	234	-5.3	N/A
Fuji IX	225	256	+13.8	N/A
Amalgomer	330	398	+20.6	N/A
Amalgomer CR	346	423	+22.3	N/A
ChemFil Superior	177.1	182.7	+3.2	n.s
ChemFlex	197.8	227.1	+14.8	0.01
Fuji IX FAST	155.7	194.4	+24.9	n.s.
Chelonfil	172.5	153.8	-10.8	N/A
Fuji II	120.5	120.5	0.0	N/A
Fuji II	210	201	-4.3	N/A
Fuji II cap	156	217	+39.1	N/A
Miracle Mix	128	166	+29.7	N/A
Ketac-Fil	172	202	+17.4	N/A
Ketac-Silver	170	208	+22.4	N/A
HiFi	210.2	200.8	-4.5	n.s

Most of the data for commercial GICs was not in a form permitting to determine the statistical significance of the difference between 24-hour and 28-day results. Therefore, an appreciable difference was arbitrarily defined as 10% or greater. Eleven materials, in addition to Aspa, showed appreciable increases, while 2 showed appreciable decreases. Six had increases similar to Aspa's *i.e.* >20%. A hypothesis was postulated when Aspa was replaced by the stronger product ChemFil, which showed no increase. It stated that strong materials had reached their maximum possible strength within 24 hours. However, the more modern materials in this survey fail to support this hypothesis. The 24-hour strength of Amalgomer CR is 1.9x that of Aspa, but this material shows almost an as high percentage increase as Aspa [3]. There is no significant correlation between 24-hour strength and change from 24 hours to 28 days (Fig. 1).

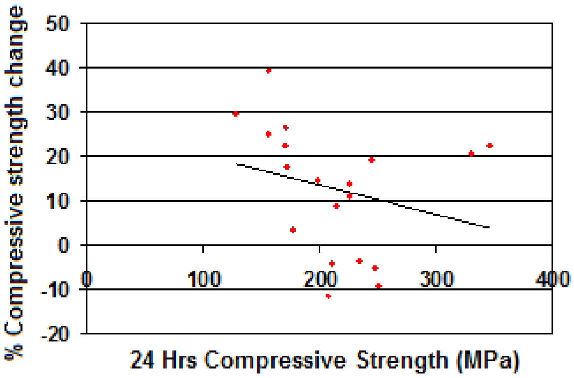

Fig. 1. Change in compressive strength of commercial restorative GICs

The correlation coefficient of the best-fit regression line is only –0.1346. Of the materials that did not show an appreciable increase, only two showed an appreciable decrease. The ten that showed no appreciable change were divided as three with slight increases, six with slight decreases and one with no change. So, of the twenty-four materials (including Aspa) with available data, twelve show appreciable increase and twelve do not. From this it can be concluded that increasing compressive beyond twenty-four hours is far from a universal property of GICs.

Dividing the data reported for restorative consistency experimental GICs into the same groups as commercial GICs, a very similar general picture emerges, as follows: seventeen of them show appreciable increases and sixteen no appreciable increases, which supports the conclusion drawn above. All experimental materials (except for ASPA III, ASPA 2005 and Na Anorthite) were formulated in the same way, *i.e*. the polymeric acid was the same (acrylic acid homopolymer of MWt ~50kD), as well as the proportions of glass: polymeric acid: water: tartaric acid. This enables us to conclude that differences in glass composition alone can give rise to differences in maturation changes in compressive strength, as also found in the commercial products ChemFil II and Opusfil W, which effectively differ only in glass composition yet show appreciable decrease and increase, respectively. In contrast to commercial materials, where 24 hour strength shows no correlation with change, a very significant inverse correlation is found (correlation coefficient = -0.859 p=<0.001) between these in experimental materials (Fig. 2).

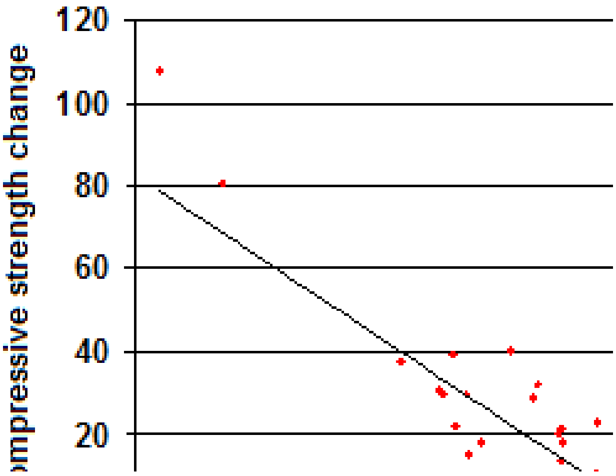

Fig. 2. Change in compressive strength of experimental restorative GICs

This correlation is somewhat influenced by the glasses with high phosphorus contents, which produce very weak and also very slow setting cements. They show very large percentage changes. However, removal of the two furthest outliers, only reduces r to -0.723, which has a p value <0.001. (It should be noted that for all experimental materials for which statistical significance can be calculated, appreciable differences are always significant and significant differences are appreciable in 27 of 33 instances for "high formulations", and 32 of 33 instances for "low formulations", respectively - Tables 2 and 3).

Table 2. Changes in compressive strength in experimental GICs (restorative)

Material	CS 24hr	CS28day	%change	Sig level
ASPA III{exp}[1]	130	182	+40.0	N/A
LG116{F exp}[2]	74..3	81.2	+9.3	0.012
LG115{F exp}[2]	165.6	165.9	+0.5	0.001
LG134{F exp}[2]	159.7	176.6	+10.6	0.001
LG26{F exp}[2]	200.6	185.3	-7.6	0.05
LG95{F exp}[2]	200.3	190.5	-6.2	0.05
LG96{F exp}[2]	201.1	193.4	-3.8	n.s.
LG97{F exp}[2]	193.0	186.9	-3.2	n.s
LG98{F exp}[2]	195.0	198.6	+1.8	n.s
LG99{F exp}[2]	190.5	185.1	-2.8	n.s
LG123/0.75 {Al:Si exp}[3]	148.1	179.5	+21.2	<0.001
LG123/0.83 {Al:Si exp[3]	142.5	145.4	+2.0	n.s
LG123/0.98 {Al:Si exp}[3]	148.1	175.0	+18.2	<0.001
LG123/1.02 {Al:Si exp}[3]	146.8	176.2	+20.0	<0.001
LG124{Al:Si exp}[3]	139.4	183.9	+31.9	<0.001
"A" 0.0{Na exp}[4]	151.0	147.0	-2.6	n.s
"A" 0.025{Na exp}[4]	152.1	153.6	+1.0	n.s
"A" 0.05{Na exp}[4]	139.0	144.3	+3.8	0.05
"A" 0.10{Na exp}[4]	148.2	168.3	+13.6	<0.001
"A" 0.15{Na exp}[4]	156.2	168.5	+7.8	<0.001
"A" 0.20{Na exp}[4]	160.0	175.4	+9.6	<0.001
"B" 0.0{Na exp}[4]	147.5	156.9	+6.4	0.05
"B" 0.05{Na exp}[4]	119.8	141.6	+18.2	<0.001
"B" 0.10{Na exp}[4]	110.9	135.1	+21.8	<0.001
"B" 0.20{Na exp}[4]	115.9	134.4	+15.0	<0.001
"B" 0.30{Na exp}[4]	109.8	152.8	+39.2	<0.001
"B" 0.45{Na exp}[4]	106.6	137.9	+29.9	<0.001

“B” 0.50{Na exp}[4]	105.4	137.3	+30.3	<0.001
-0.5{P exp}[5]	160.0	196.6	+22.9	<0.001
0.0{P exp}[5]	191.1	185.3	-3.0	n.s
0.2{P exp}[5]	133.3	146.6	+10.0	<0.001
0.4{P exp}[5]	91.8	126.4	+37.7	<0.001
0.6{P exp}[5]	29.9	54.1	+80.5	<0.001
0.8{P exp}[5]	8.3	17.3	+107.9	<0.001
Na Anorthite[6]	115.0	149.0	+29.6	N/A
“ASPA 2005”[7]	138	178	+29.0	

Table 3. Compressive strength changes in experimental GICs (luting)

LG116 {F exp}[1]	42.8	52.6	+22.9	<0.001
LG115 {F exp}[1]	73.4	86.8	+18.3	<0.001
LG134 {F exp}[1]	90.2	95.8	+6.2	0.05
LG26 {F exp}[1]	101.4	101.5	+0.1	n.s
LG95 {F exp}[1]	90.9	110.8	+21.9	<0.001
LG96 {F exp}[1]	85.7	99.8	+16.5	<0.001
LG97 {F exp}[1]	71.3	104.2	+46.1	<0.001
LG98 {F exp}[1]	77.2	98.8	+28.0	<0.001
LG99 {F exp}[1]	87.3	97.6	+11.8	<0.001
LG123/0.75{Al:Si exp}[2]	77.0	97.3	+26.4	<0.001
LG123/0.83{Al:Si exp[2]	59.0	89.7	+52.0	<0.001
LG123/0.98{Al:Si exp}[2]	69.8	85.9	+23.1	<0.001
LG123/1.02{Al:Si exp}[2]	59.5	73.0	+22.6	<0.001
LG124{Al:Si exp}[2]	63.7	75.6	+18.7	0.002
“A” 0.0{Na exp}[3]	76.7	84.9	+10.7	<0.001
“A” 0.025{Na exp}[3]	69.0	94.4	+38.8	0.001
“A” 0.05{Na exp[3]	61.0	77.4	+26.9	0.001
“A” 0.10{Na exp}[3]	67.0	96.5	+44.0	0.001
“A” 0.15{Na exp}[3]	76.3	93.6	+22.7	0.002
“A” 0.20{Na exp}[3]	70.5	100.5	+42.6	0.001
“B” 0.0{Na exp}[3]	67.4	85.4	+26.7	<0.001
“B” 0.05{Na exp[3]	70.2	74.9	+6.6	n.s
“B” 0.10{Na exp}[3]	56.4	79.2	+40.4	<0.001
“B” 0.20{Na exp}[3]	57.9	85.1	+47.0	<0.001
“B” 0.30{Na exp}[3]	65.5	83.3	+27.2	<0.001
“B” 0.40{Na exp}[3]	61.5	80.6	+31.1	<0.001
“B” 0.50{Na exp}[4]	59.2	90.7	53.2	<0.001
-1.5{P exp}[4]	63.2	64.0	+1.3	n.s
-0.5{P exp}[4]	73.7	101.7	+38.0	<0.001
0.0{P exp}[4]	101.4	101.5	+0.1	n.s
0.2{P exp}[4]	84.6	87.9	+3.9	n.s
0.4{P exp}[4]	68.1	79.8	+17.2	<0.001
0.6{P exp}[4]	43.5	55.7	+28.0	<0.001
0.8{P exp}[4]	12.5.	25.6	+104.8	<0.001

An obvious influence on compressive strength change is formulation. In all tests reported in papers elaborated at Limerick University [4-8], the cements were tested both as “high formulation” and “low formulation”. These two cement formulations studied are shown in Table 4, where the low formulation contains more water and tartaric acid than the high one. (Note that all components, *i.e.* glass, polymeric acid and tartaric acid, are the same. The high formulation is similar to that of a restorative dental GIC and the low one to a luting GIC. However, most commercial luting cements have lower glass: polyacid ratios, as well higher water and tartaric acid contents than equivalent restoratives.)

Table 4. Changes in the formulation of experimental GICs [Wt% & %]

	Glass	Polyacid	Water	Tartaric acid
Hi Formulation	70.9	14.2	13.4	1.5
Lo Formulation	66.0	13.2	18.7	2.1
Change Hi to Lo	-6.9	-7.0	+39.6	+40.0

Overall, as shown in Table 5, changing their proportions gives a mean Δ CS for “low formulation” twice as high as for “high formulation”. Additionally, for the experimental glasses tested as both formulations, the high formulations showed appreciable increases in 17 of 33 cases, whereas this occurred in 27 of the low formulation examples. However, the value of the correlation coefficient for CS_{24} v Δ CS for all cements is only slightly lower than for the “high formulation”, compared to the “low formulation” (r = -0.707 compared to -0.859).

Table 5. Mean percentage increase/ decrease in CS 1 to 28 days for high/ low formulations

Experiment	Low	High
Δ P	+17.8	+18.2
Δ F	+18.1	-1.2
Δ Al:Si	+27.2	+18.0
Δ Na(Glass"A")	+30.1	+5.6
Δ Na(Glass"B")	+33.2	+22.2
Overall Mean	+25.2	+12.5

In two of the experimental runs where the F content was changed and one where the Na content of "A" type glass was changed, low formulations showed appreciable increases, whereas the equivalent high formulations did not.

There are three factors that might be related to formulation change and potentially to CS change. The additional water content might enable changes in the polyacid/ cross-linking cations interactions to continue for longer times. Secondly, water might change from unbound to bound water to a greater extent [9]. Thirdly, the higher tartaric acid content might result in strongly crosslinking ions, *i.e.* Al^{3+} being exchanged from tartarate to carboxylate groups on the polymer increasing the crosslink density [10]. It should be noted that the 24 hour CS of low formulations was much lower that that of the high formulation. In addition, the setting time of low formulation GICs is normally much longer than the equivalent high formulation. However, an experiment designed to evaluate accelerated setting of the same GIC with two types of external radiation [11] did not support this hypothesis. Although radiant heat increases appreciably the 24 hour compressive strength compared to the material allowed to self cure, the ΔCS values are very similar - see Table 6. Also, the commercial GIC used in the study (Fuji IX Fast) has one of the shortest setting times of all such cements, yet showing an almost identical Δ CS value to that of the very slow setting Aspa: +24.8% and +26.5%, respectively.

Table 6. Effect of external radiation on CS change from 24 hours to 28 days

Cure Type	CS 24hr	CS 28 days	Δ CS ± %
Self Cure	155.7	194.4	+24.8
Ultrasound	188.8	218.3	+15.6
Radiant Heat	193.3	240.1	+24.2

When GICs are stored in water or oil at a temperature range from 23 to 70C, the CS change is generally reduced as temperature increases [12] (Fig. 3).

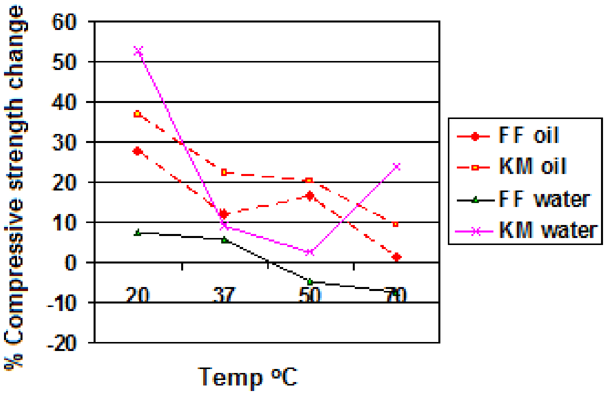

Fig. 3. Changes in temperature versus compressive strength

In summary, it appears that different glass compositions influence the extent of CS change with maturation. Changing the cement composition by increasing the water and tartaric acid levels increases Δ CS for cements, in some but not in all experimental glass series. For commercial cements there is no significant association between 24 hour CS and Δ CS whereas, for experimental GICs, there is a highly significant negative correlation.

Considering all the possible explanations for the increase in compressive strength, namely:

Replacement of non-crosslinking carboxylate groups (*i.e.* those neutralised by Na or other monovalent cation) does not appear to be an explanation, since the majority of experimental glasses are free of Na and other monovalent cation formers. Change of the Na content in the "B" series does show some association between an increasing Na content and increase in Δ CS, but this is not shown by the "A" series (Tables 2 and

3). All commercial restorative GICs contain Na in varying amounts, but there appears to be no correlation between Na content and Δ CS. In particular, both ChemFil and Opusfil, which show appreciable changes in CS in opposite directions (see above) have almost identical Na levels in their glasses - of 6.3% and 5.5%, respectively.

OTHER STRENGTH MODALITIES

As stated above, most measurements of strength changes are made in compression, since this has been used historically and in all test standards. Uni-axial tensile strength was examined for some of the GICs generation that replaced Aspa [13], but the compressive strengths of these materials [3] increases were not universally found. The diametral tensile strength has been used more often than the more appropriate flexural strength but, again, only few investigators evaluated the effect of maturation time. The results obtained by Mitra *et al.* [14] enable a comparison between the changes in diametral and compressive strength and those in compressive modulus (Fig. 4). There appears to be a very loose association between the changes in these three modalities, seemingly related to low initial [24hr] strengths, and resulting in large changes.

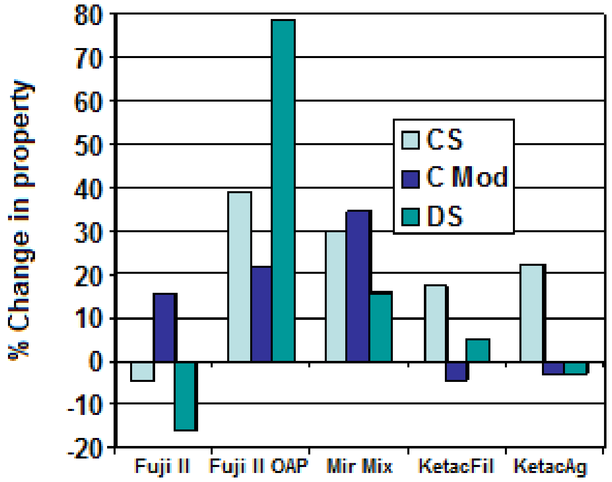

Fig. 4. Changes in compressive and diametral strength and compressive modulus

An interesting result was obtained when comparing biaxial flexural strength changes in two GICs that only differed in the particle size of the glass component [15]. The experimental cement with a broad particle size distribution increased by 30.0% from one to twenty eight days, whereas the commercial version, with a narrower distribution, decreased by 12.5%. These changes were shown in samples stored in water; those stored in artificial saliva behaved similarly.

OTHER MECHANICAL PROPERTIES

Hardness was measured in the original paper of Crisp *et al.*, who reported maturation changes in a GIC [1]. Little information was actually available. A more recent paper of de Moor *et al.* [16] reported considerable differences between restorative commercial GICs materials stored in air and in water. Although all ten materials showed considerable increases from the 15 minute values (when de-moulded) when stored in water or humid air for 1 day, all but one of the water stored ones showed decreases from this value after one month. In contrast, the humid air samples showed marked increases, with one exception (Fig. 5).

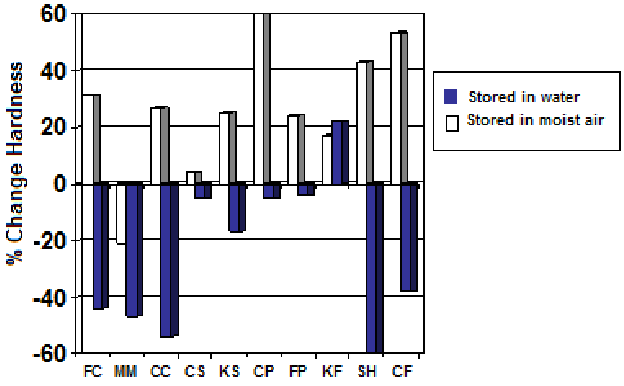

Fig. 5. Hardness changes 24 hours to 1 month

A study of Griffin and Hill [17] compared the effects of changing molecular weight of the polymeric acid and the fluorite content of glass on flexural strength, Young's modulus, toughness, and fracture toughness. As Tables 7-9 show, neither modality uniformly affects change with age in any of the four properties tested. However, flexural strength shows a mean increase of 32%, and 13 of 16 results show appreciable increases. The modulus shows a mean increase of 69%, and all 16 results increase appreciably. Fracture toughness shows a mean increase of 17%, while 13 of 16 show appreciable increases. In contrast, toughness results (not shown as a table) appreciably decreases in 14 cases and only slightly increases in 2.

Table 7. Changes (%) in flexural strength from 24 hrs to 1months; effect of molecular weight (E No) and CaF_2 content (X)

X	E5	E7	E9	E11
0	+47.7	+30.2	+34.5	+42.2
1	+8.1	+12,1	+75.2	+38.4
2	+45.2	-4.9	+4.4	+48.4
3	+36.6	13.4	+22.1	+58.6

Table 8. Changes (%) in Young's Modulus from 24hrs to 1months; effect of molecular weight (E No) and CaF_2 content (X)

X	E5	E7	E9	E11
0	+97.9	+95.6	+66.1	+51.7
1	+45.3	+40.9	+67.3	+45.7
2	+91.3	+68.0	+74.6	+63.5
3	+82.4	+27.4	+115.1	+91.1

Table 9. Changes (%) in fracture toughness from 24 hrs to 1months; effect of molecular weight (E Number) and CaF_2 content (X)

X	E5	E7	E9	E11
0	+29.6	+10.6	-12.3	+23.0
1	+2.4	+1.9	+13.0	+15.3
2	+32.1	+32.4	+9.8	+18.3
3	+20.0	+25.0	+34.1	+11.8

Stress relaxation has not been extensively reported in a pilot study, yet it appeared that it showed little change beyond 24 hours [18], whereas resin-modified GICs stress relaxation continued to reduce for longer periods [19].

ADHESION

It has not been studied over differing maturation times, although a preliminary study did indicate that adhesion increased for one GIC [20]. Since GICs on enamel and particularly on dentine appear to fail cohesively, GICs that increase in strength would be expected to show increased bond strength with time. This is in line with the findings of Algera *et al*. on the increase in bond strengths produced by application of heat or ultrasound to glass ionomers [21].

EROSION

Five GICs have been evaluated using the lactic acid jet test, which gives better correlation with *in vivo* erosion than other tests [22]. Results showed that all five GICs tested improved significantly when maturation is increased from 24 hours to 2 months (Table 10), even those that do not show strength increases [23]. Other cements tested, zinc cement, zinc polycarboxylate, and dental silicate showed no significant changes.

Table 10. Changes in Lactic Acid Jet Test Erosion with time

Material	Erosion after 24 hours (mg/day)	Erosion after 2 months (mg/day)	% change
Aspa	1.48	1.15	-22.3
Ketac Fil	1.00	0.56	-44.0
ChemFil II	0.38	0.21	-44.7
Opusfil W	0.34	0.08	-76.5
Fuji II R	0.20	0.10	-50.0

ABRASION

Like erosion, resistance to abrasion as measured by the ACTA wear test showed improveemts in all commercial restorative GICs [24-26] (Figs. 6-7). In Figure 6, the changes from 1 week to 4 months are shown as storage at 23°C, as contrasted to other modalities tested whereas, in Figure 7, the wear rates at 1 day and 1 months were compared as storage was at 37°C. In all these studies, reduction in wear was considerable. Only one glass ionomer (Ketac Silver) had a decrease in wear less than 30%.

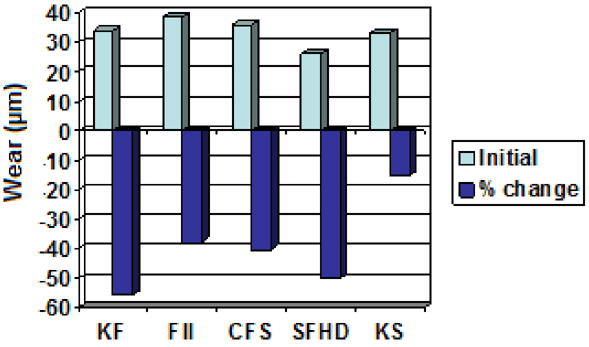

Fig. 6. Changes in ACTA wear rate at room temperature

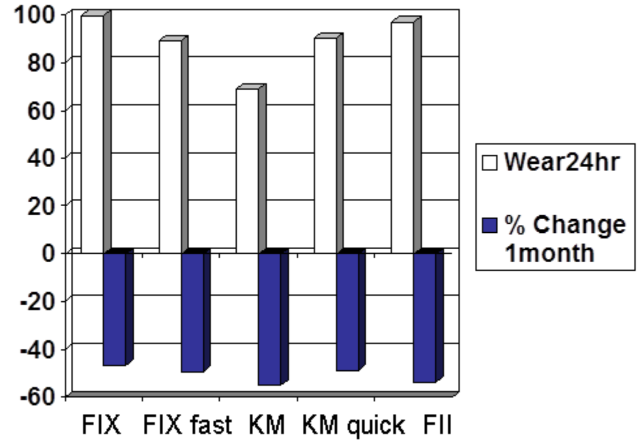

Fig. 7.Changes in ACTA wear rate at 37°C

VISUAL OPACITY

Aspa was extremely opaque compared to DSC and composite resin restorative. It was reported that "translucency improved with maturation", but the changes observed [27] were small, compared to DSC. Much of the opacity of Aspa was due to the phase separation of glass, rendering it opaque and consequently making unlikely maturation changes in cement opacity. More modern GICs have lower levels of opacity [28-30] but little has been reported on opacity changes over longer term for them. However, Yip *et al*. reported in 2002 that the colour *in vivo* improved for two GICs over one year [31].

4. CONCLUSIONS

It is clearly established that glass ionomers can improve appreciably in strength from 1 to 28 days, but that is not a universal property. Changes in the chemical composition of the glass component can produce a different behaviour in this respect, as the differences in particle size distribution.

In contrast, the clinically important resistance to both erosion and abrasion improves with maturation for all of the limited number of GICs tested, even for those that showed no increase in strength.

Variation of the effect of maturation on different properties may be related to changes in E elastic modulus and toughness G1, with time. Increasing crosslink density between carboxylate groups with time would be expected to result in increases in modulus E, but not necessarily in toughness G1. This difference would account for the "universal" improvements observed in resistance to abrasion and erosion, which are expected to increase with crosslink density. For strength, however, increase in E may be counterbalanced by reductions in toughness.

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