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Evaluation and comparison of physical properties and fluoride release of newly introduced ceramic reinforced glass-ionomer restorative material with other glass ionomer cements – An *in vitro* study

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Abstract
Aim : The purpose of the study was to evaluate and compare the fracture toughness, compressive strength, flexural strength and check the amount of fluoride release of Amalgomer CR, Ketac N100, DyractXtra and Giomer dental restorative material.
Methodology: 100 specimens will be used in 4 different groups of 25 each (Amalgomer CR, Ketac N 100, DyractXtra and Giomer). The physical properties (fracture toughness, compressive strength, flexural strength) will be evaluated using Universal Testing Machine and amount of fluoride release using Fluoride ion specific electrode from the above mentioned glass ionomer cements.
Results: Amalgomer CR showed the highest compressive strength and # Toughness compared to other GIC material used for the study, however the Flexural Strength of Giomer was found to be highest compared to Amalgomer CR and other GIC material used in my study. In terms of Fluoride releasing capacities Amalgomer CR showed increased fluoride release for the first 24 to 48 hr reaching a peak at 6th hr, thereafter decreasing slowly to reach steady level at fifth week.
Conclusion: Within the limitations of my study it could be stated that Amalgomer CR can be used as an effective restorative material due to its higher physico-mechanical properties and fluoride releasing capacities as compared to various other GIC restorative materials used in the study.

Keywords: Fracture Toughness, Compressive Strength, Flexural Strength, Fluoride Release, Universal Testing Machine, Fluoride ion specific electrode, Amalgomer CR, Ketac N 100, DyractXtra and Giomer

1. Introduction

Caries is a dynamic process in which mineral is removed during times of high acid production by bacterial plaque (demineralization) and replaced during periods of neutral pH (re-mineralization). Fluoride has been well documented as a major contributing factor for the decline in the incidence and severity of dental caries and plays a central role in prevention of dental caries [1].

Today, there are several fluoride-containing dental restoratives available in the market such as glass-ionomers, resin modified glass-ionomer cements, polyacid modified composites, composites and giomers. Due to their different matrices and setting mechanisms, the products vary in their ability to release fluoride. The use of restorative materials with the highest long-term fluoride release is preferable, especially in patients with moderate-to-high caries activity. The exact minimal fluoride concentration for caries inhibition has not been determined [1].

Glass ionomer cements (G1C) possess certain unique properties like release of anti-cariogenic fluoride into adjacent tooth structures, chemical bonding to enamel and dentine and a low coefficient of thermal expansion similar to tooth. They are, however, susceptible to fracture and exhibit low wear-resistance. These deficiencies have limited their use and made them unsuitable for high-stress areas such as class I and II restorations. Because of their low tensile strength, fracture toughness and brittleness, a variety of modifiers have been added to conventional glass ionomers, to improve their mechanical properties. These have included changing the composition-for example, the fluoride and sodium content and the aluminium:

silica ratio; adding 'bioactive'components such as certain glasses and hydroxyapatite; and reinforcement by incorporating metal particles such as silver–tin alloy, gold, platinum, palladium, stainless steel or fibers such as carbon, steel or glass [2].

In the late 1980´s, the addition of polymerizable hydrophilic resins to conventional glass ionomer cements resulted, in the development of resin-modified formulas that set by a dual reaction: the acid–base reaction and a free radical polymerization process. In general, resin modified glass ionomer cements were reported to show better mechanical properties than conventional glass ionomers, even though there are individual differences from one brand to another. Still their polymerization shrinkage and low wear resistance constitutes a major drawback [3].

Recently, a new ceramic-reinforced glass ionomer (Amalgomer CR) has been introduced to the dental market. This tooth-colored product is proposed by the manufacturer to combine the high strength of a metallic restorative and the esthetics and other advantages of glass ionomers [3].

The purpose of this *in vitro* study is to evaluate the fracture toughness, compressive strength, flexural strength and fluoride release of newly introduced Amalgomer CR with Ketac N 100, DyractXtra and Giomer dental restorative material (Fig. 1).

Fig 1: Materials used (Amalgomer CR with Ketac N 100, DyractXtra and Giomer [Beautifil II])

2. Materials and methods

A total of 100 specimens were equally divided into 4 groups of 25 in each, out of which 15 specimens in each group were used for the evaluation and comparison of physical properties and 10 specimens from each group were used to evaluate and compare the amount of fluoride release from four glass ionomer cements.

Sampling method: Random sampling method will be used for the study.

Statistical method used: 1) ANOVA test; 2) Post hoc turkey test; 3) Fosher Exact test

1) Fracture toughness

Twenty knife-edge notch specimens (25 mm length _ 2.5 mm thickness _ 5 mm width) were prepared in a stainless steel split mould (n = 5) (Fig. 2). The GI powders of each group was mixed according to the manufacturer's instructions. After that, the mould was filled by the mix and covered with two matrix strips and glass slides to avoid pore incorporation. After setting the specimens was removed from the mould and the flash removed by grinding on wet 600-grit silicon carbide (SiC) abrasive paper. The specimen was then stored in distilled water at 37 ^{0}C for 24 h prior to testing.

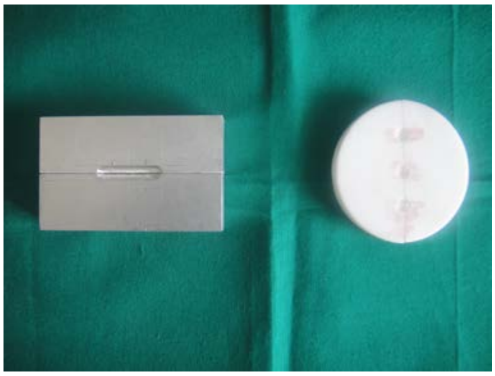


Fig 2: Stainless Steel Mould and Teflon Mould

Fig 3: Strength Check Sample.

The fracture toughness was determined according to ASTM standard E-39924 and the specimen was subjected to three-point bending on a universal testing machine at a crosshead speed of 0.5 mm/min. Fracture toughness, K1c (MPa m1/2), was determined according to the following formula:

$$K_{1c} = F\left(\frac{a}{w}\right)\left(\frac{PQ^S}{BW^{3}/_{2}}\right)$$

where P is the peak load (kN), s is the span length between supports (cm), B is the specimen thickness (cm), W is the specimen width (cm) and a is the crack length (cm) and f (a/W) is a function of a/W and was calculated as:

$$3\sqrt{(a\,/\,W)}[1.99 - (a\,/\,W)$$

$$F\left(\frac{a}{w}\right) = \frac{(1 - {}^{a}/_{W})\{2.15 - 3.93\,{}^{a}/_{W} + 2.7[{}^{a}/_{W}]^{2}\}}{\dfrac{2\left(1 + \frac{2a}{W}\right)\left(1 - \frac{a}{W}\right)3}{2}}$$

2) Flexural strength

Twenty bar-shaped specimens (25 mm length _ 2 mm thickness _ 2 mm width) (Fig. 5) were prepared in rectangular-shaped stainless- steel split mould as described for the fracture toughness test above.

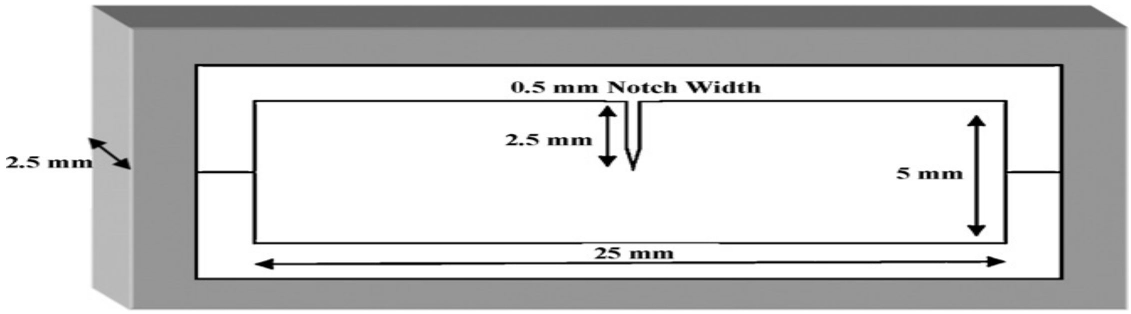


Fig 5: Schematic diagram of specimen preparation for fracture toughness test. Stainless steel split mould (25 mm length T 2.5 mm thickness T 5 mm width) with 0.5 mm notch width and 2.5 mm depth surrounded by a frame.

The flexural strength test was performed based on ISO standard and it was subjected to a three-point bending in a universal testing machine at across head speed of 0.5 mm/min. Flexural strength, s (MPa), was calculated using the following formula:

$$\sigma = \frac{3Pl}{2bd^2}$$

where P (N) is the load at fracture, l is the distance between the two supports (mm), b is the width of the specimen (mm) and d is the thickness (mm).

3) Compressive strength

Twenty cylindrical specimens were prepared in a stainless steel split mould (4 mm in diameter and 6 mm in height) according to ISO standard. The compressive strength (MPa), Cs, of the specimens will be performed using the universal testing machine (Fig 6) at a crosshead speed of 0.5 mm/min and is calculated using the following equation:

$$C_S = \frac{4P_f}{\pi D^2}$$

where Pf is the load (N) at fracture and D is the diameter of specimen (mm).

Fig 6: Universal Testing Machine (UTI)

4) Fluoride release

The study was done using four types of fluoride-releasing restorative materials, which were commercially available at the time of study. The materials selected were divided into four groups as follows: A total of 40 specimens, with 10 disk specimens for each group, were prepared. The materials were manipulated as mentioned earlier. Specimens were prepared by filling the custom-made Teflon mould (diameter of 5 mm, depth of 2 mm) (Fig.4) and Mylar strip was placed on the surface of the specimen and pressure was applied to extrude excess material.

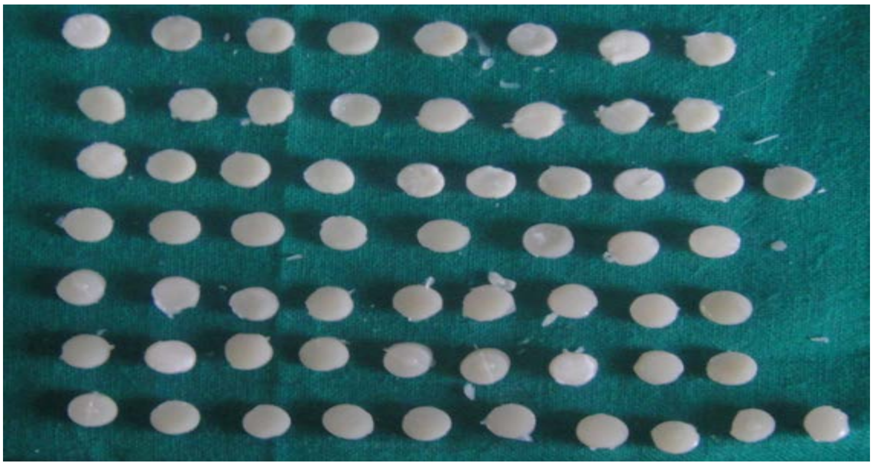


Fig 4: Fluoride Release Sample

Immediately after setting, each specimen was immersed in an individual polyethylene tube with 5 ml of artificial saliva. The fluoride estimation was done at 6h, 24h, 48h and weekly intervals for 5 weeks.

3. Results

Table 1: Mean Physical Properties of GIC materials

Group	# toughness (mpa $m^{1/2}$)	Compressive strength (mpa)	Flexural strength (mpa)
Amalgomer CR	0.826 MPa $m^{1/2}$	342.5 MPa	73.5 MPa
Ketac N 100	0.599 MPa $m^{1/2}$	252.3 MPa	53.4 MPa
Dyract Xtra	0.616 MPa $m^{1/2}$	315.9 MPa	71.6 MPa
Giomer	0.566 MPa $m^{1/2}$	324.4 MPa	81.7 MPa

In Table 1 Amalgomer CR shows the highest compressive strength of 342.5 MPa compared to other GIC materials tested in the study. Amalgomer CR shows the highest # Toughness of 0.826 MPa $m^{1/2}$ compared to other GIC materials tested in the study. Giomer (Beautifil) shows the highest Flexural Strength of 81.7 MPa compared to Amalgomer CR (73.5 MPa) and other GIC materials tested in the study.

Table 2: Fluoride Release (ppm) From Four Groups During Test Intervals Up to 5 Weeks.

Group	6 h	24 h	48 h	1 week	2 week	3 week	4 week	5 week
Amalgomer CR	1.48	1.42	1.38	1.31	1.31	0.87	0.83	0.75
Ketac N 100	0.75	0.83	0.65	0.53	0.34	0.31	0.34	0.32
Dyract Xtra	0.33	0.67	0.78	0.92	0.73	0.64	0.65	0.61
Giomer	0.39	0.63	0.91	0.98	1.01	0.80	0.67	0.65

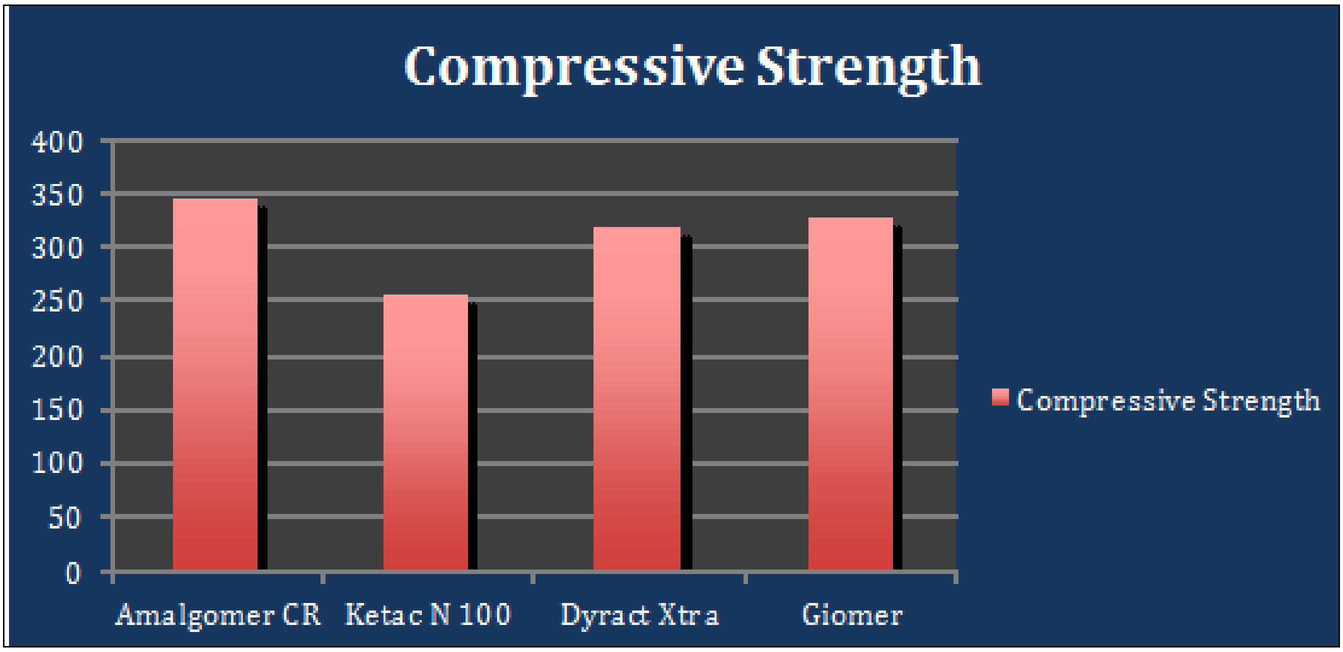
Table 2 shows Fluoride Release (ppm) From Four Groups During Test Intervals Up to 5 Weeks. Amalgomer CR shows increased fluoride release for the first 24 to 48hr reaching a peak at 6^{th}hr, thereafter decreasing slowly to reach steady level at fifth week. Ketac N 100 showed increased fluoride release at 6^{th} hr compared to Dyract Xtra and Giomer but showed decreased fluoride release compared to Amalgomer CR with a peak at 24^{th} hr. DyractXtra released the least amount of fluoride among the tested materials with peak reaching at 1^{st} week (0.92ppm). Giomer showed increased fluoride release for the first 2 weeks reaching a peak at 2^{nd} week (1.01), thereafter decreasing slowly to reach steady level at fifth week.

Table 3: The Mean Fluoride Release (ppm) From Four Groups of GIC material.

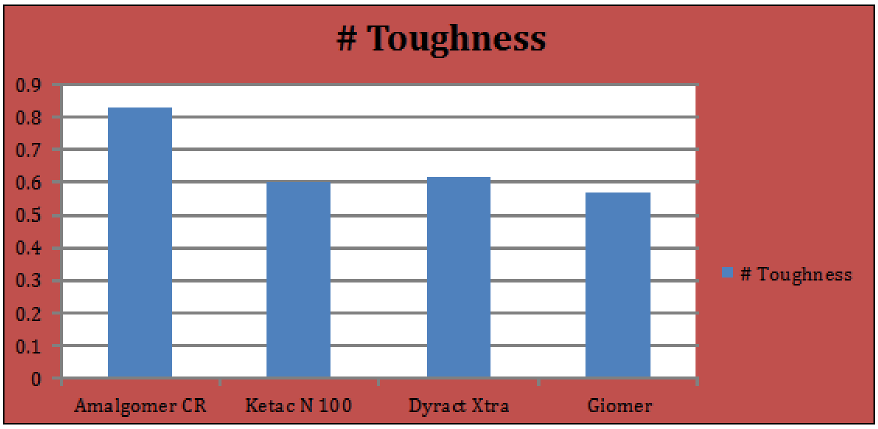
	Amalgomer CR	Ketac N 100	Dyract Xtra	Giomer
Fluoride Release	1.168 ppm	0.508 ppm	0.666 ppm	0.755 ppm

Table 3 shows Mean Fluoride Release (ppm) From Four Groups During Test Intervals Up to 5 Weeks. Amalgomer CR shows the highest Mean Fluoride release among all the tested GIC products used in the study.

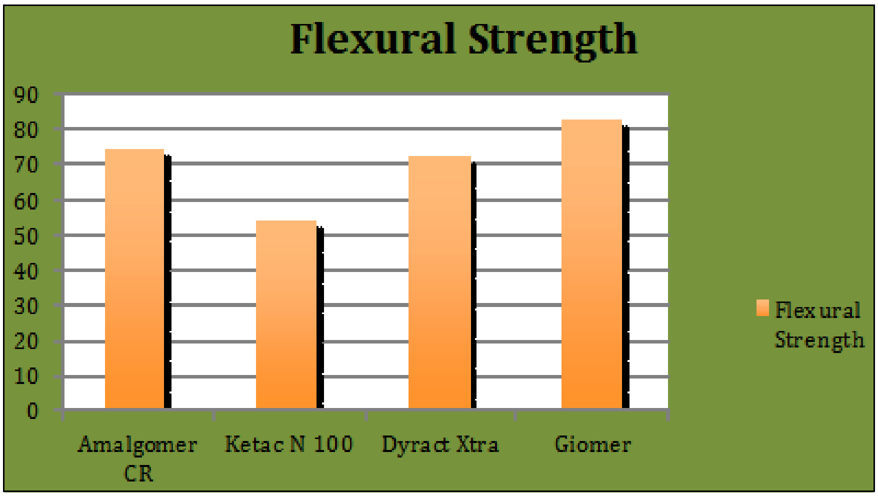
3.1 Plots of Means Section



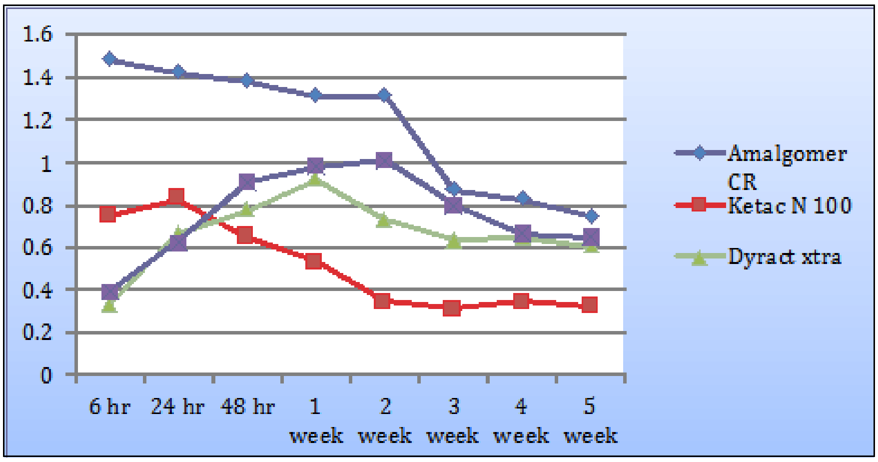
Graph 1: The Mean Compressive Strength of GIC material



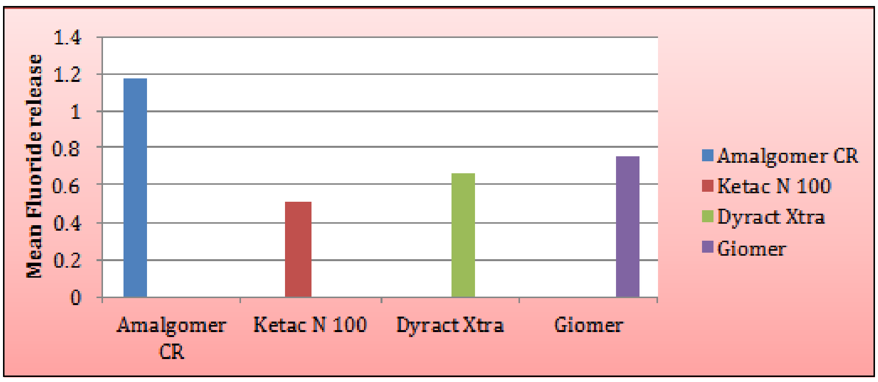
Graph 2: The Mean # Toughness of GIC materials



Graph 3: The Mean Flexural Strength of GIC material



Graph 4: Fluoride Release (ppm) From Four Groups During Test Intervals Up to 5 Weeks.



Graph 5: The Mean Fluoride Release (ppm) From Four Groups of GIC material.

4. Discussion

Recently, a new ceramic-reinforced glass Ionomer (Amalgomer CR) has been introduced to the dental market. This tooth-coloured material as claimed by manufacturer combines the high strength of a metallic restorative, esthetics and other advantages of glass-ionomers. Amalgomer CR is ceramic reinforced GIC which not only complies with the international standards of GIC but with the standard for amalgams. The ceramic also helps in imparting excellent wear and erosion resistance and also enhances the radiopacity and all round strength of the cement [4].

According to its setting mechanism, Amalgomer CR is a conventional acid–base reaction GIC. The product includes a particulate ceramic component with the intention of increasing its strength, supposedly without sacrificing appearance (although it is opaque white) or other general characteristics of GIC. It has been shown elsewhere that zirconia is the major if not the only (crystalline) component of the additive of this product, similar to that of other laboratory studies done by Gu YW, Biomaterials 2005; 26(7):713–20. Zirconia is known to be an excellent material for strengthening and toughening in certain composite contexts because of its peculiar character of a phase transformation from tetragonal to monoclinic under stress. This transformation produces a 4% change of volume which generates a local compressive stress, which then offsets crack-opening tension and so inhibits crack propagation and increasing the incorporating material's fracture resistance. This effect in otherwise very brittle ceramics may have prompted its use in GIC, although it is not known that it would function in this manner in the more ductile matrix. In addition, the manufacturer claims that the ceramic filler is able to react partially with the matrix, which may produce some bonding (and so matrix constraint) and also possibly an altered polysalt matrix [2].

In the present study, the temperature was kept equal to the normal body temperature by placing the samples in an incubator at 37 °C. Deionized water was chosen for the experiment as it provided the baseline of fluoride release potential in unstimulated conditions. This is in agreement with the earlier studies [4].

Artificial saliva was chosen as a second medium for fluoride leaching so as to simulate to an extent the natural oral environmental conditions, although, duplicating exactly the properties of human saliva is impossible due to the inconsistent and unstable nature of natural saliva. So the development of artificial saliva is essential for well justified and controlled experiments. The present study utilized fluoride ion-specific elecrode (Orion, 94098N) and microprocessor ion analyzer (Orion, 960) since it is simple and convinient method [4].

Fluoride Release (ppm) from Four Groups during Test Intervals Up to 5 Weeks, Amalgomer CR shows increased fluoride release for the first 24th to 48thhr reaching a peak at 6thhr, thereafter decreasing slowly to reach steady level at fifth week. The coarse ceramic particles reinforced in glass ionomer of Amalgomer CR may contribute to its high fluoride release. This is supported by DeSchepper and Others (1991) who observed that the coarse silver alloy particles in Argion (a metal reinforced glass ionomer), which are not bound to the cement matrix, result in an increase in the microporosity of the cement, thus increasing the effective surface area available for elution of fluoride [4]. Ketac N 100 showed increased fluoride release at 6th hr compared to Dyract Xtra and Giomer but showed decreased fluoride release compared to Amalgomer CR with a peak at 24th hr. This could be due to "Nanomers" which are small sized particles which plays a significant role in this regard. The small glass particles of Ketac N 100 provides a larger surface area which increases the acid base reactivity and hence has the capacity to release fluoride from the powder more quickly, increasing the fluoride release of the material. However it showed low fluoride release as compared to the other two materials. This phenomenon could be explained by its low solubility [5].

DyractXtra released the least amount of fluoride among the tested materials with peak reaching at 1st week (0.92ppm). This result is in agreement with the findings of Forsten (1995); who compared Dyract cem with other products. Giomer showed increased fluoride release for the first 2 weeks reaching a peak at 2nd week (1.01), thereafter decreasing slowly to reach steady level at fifth week [5].

Giomer (Beautifil) showed little amount of controlled fluoride release in study. Giomer (Beautifil) contains Surface Pre Reacted Glass Ionomer (S-PRG) as a fluoride component. The fluoride glass within Giomer (Beautifil) has little or no glass ionomer matrix phase, because of the lack of any significant acid base reaction. As PGR has been pre reacted with fluoroalumisilicate glass and acid, water sorption is not critical in the acid base reaction as seen in this study and is in agree with results of other studies (Yap *et al*. 2002; Itola *et al*. 2004b) [6].

It is important to note that although this study was designed to mimic daily occurrences of acid challenges and fluoride exposures seen with typical homecare regimens, *in vitro* results may not be directly representative of *in vivo* results. Fluoride release measured from specimens immersed in a static medium may not take into account the dynamic nature of conditions found in the oral cavity. Although the majority of fluoride release studies are designed in this manner, there are some that attempt to more closely simulate intraoral conditions [7].

Further *in-vivo* studies are needed to evaluate and compare the physical properties and fluoride release of this newly introduced Amalgomer CR with other commonly used restorative materials in clinical practice.

5. Conclusion

Amalgomer CR showed the highest compressive strength and # Toughness compared to other GIC materials tested in the study. Giomer (Beautifil II) however showed the highest Flexural Strength compared to Amalgomer CR and other GIC materials tested in the study. In terms of Fluoride release Amalgomer CR showed increased fluoride release for the first 24 to 48 hour reaching a peak at 6th hour, thereafter decreasing slowly to reach steady level at fifth week. In terms of Mean Fluoride Release (ppm) From Four Groups during Test Intervals Up to 5 Weeks, Amalgomer CR shows the highest Mean Fluoride release among all the tested GIC products used in the study.

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