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THE EFFECT OF THREE DIFFERENT P/L RATIO ON THE TENSILE AND SHEAR BOND STRENGTHS OF MANUALLY MIXED GIC

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ABSTRACT

Purpose: To evaluate the effect of there different P/L ratios on the tensile and shear bond strengths of Manually Mixed GIC.

Materials and method: A total of 28 upper maxillary molar resin dies were constructed, on which metal copings –with loops- were fabricated. Samples were divided into four groups according to P/L ratio of GIC cement used (Ketac-Cem, 3M ESPE, USA), for coping cementation; group A; 1 scoop/1 drop, group B; 1 scoop/2 drops (control), group C; 1.5 scoops/1 drop and group D; 2 scoops/1 drop. Samples were submitted to a tensile test using a universal testing machine (Instron 8874; Instron Corp. Canton, Mass) under a constant crosshead speed of 1.00 mm/min. Another 28 upper maxillary molar were embedded in transparent acrylic resin (Vertex- Dental B.V., The Netherlands). The blocks were trimmed to expose dentine surface on which a 3x3 mm Cr-Co cubes with a thickness of 2 mm were cemented on them. Samples were also divided according to P/L ratio used. A mono-bevelled chisel shaped metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5 mm/min was used to apply load on the side metal cubes attached to lower compartment, for shear bond testing. All data was collected and statistically analyzed.

Results: The highest mean value was observed in group C (409.25 MPa) and the lowest mean value was observed for group A (235.69 MPa). Statistical analysis revealed significant difference between groups (P= 0.010). Statistical analysis among groups showed significant difference between groups A and C (P= 0.017), between groups B and C (P= 0.003) and between groups B and D (P= 0.033). But no significant differences were found between groups A and B (P= 0.879), groups A and D (P=0.062) or between groups C and D (P= 0.731). (Table 3) Regarding shear bond strength, the highest mean value was observed in group A (8.39MPa) and the lowest mean value was observed for group B (1.06 MPa). Statistical analysis revealed significant difference between groups (P= .000). Statistical analysis among groups showed significant difference between groups A and B (P= 0.000), between groups A and C (P= 0.002), between groups A and D (P= 0.001) and between groups B and D (P= 0.032). But no significant differences were found between groups B and C (P= 0.063), or between groups C and D (P= 0.870).

Conclusions: Within the limitations of the study it could be concluded that, the increase in the P/L ratio to 1.5 scoops/1 drop or 2 scoops/1 drop improved tensile and shear bond strengths Of hand mixed GIC than the manufacturer's recommendation. The 1 scoop/1 drop group improved shear bond strength but not tensile strength of hand mixed GIC than the manufacturer's recommendation.

Keywords: GIC, Cement, P/L ratio, Shear bond strength, Tensile strength.

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INTRODUCTION

Proper selection of a luting agent is a last vital decision in a series of steps that will determine the long-term success of fixed restorations. One hundred years ago this decision was easy with the availability of essentially only one luting agent, zinc phosphate cement. Currently, a plethora of luting agents is available. Now the choice of the optimal luting agent can be confusing, even for the most experienced clinician. Restorations of metal, porcelain fused to metal, low-and high-strength ceramics, full or partial coverage, require a prudent approach and the proper cement selection should be based on knowledge of physical properties, biological properties and other attributes of both restorative materials and luting agents.⁽¹⁾

Glass ionomer cements (GICs) were invented in the late 1960s in the laboratory of the Government Chemist in Great Britain and were first reported on by Wilson and Kent in 1971.⁽²⁰⁾ Over the last four decades, glass ionomer cements (GICs) experienced a period of constant technological advancements⁽³⁻⁷⁾, thus becoming an excellent dental material preventive and even restorative option in some specific situations.^(4,8,9)

Glass ionomer cements (GICs) are recognized due to their remarkable advantages, such as chemical bonding ability to enamel, dentin and base metal⁽¹⁰⁻¹²⁾ and long-term fluoride release.⁽¹³⁻¹⁵⁾ Along side biocompatibility and low coefficient of thermal expansion.^(12,16,17) However, these cements have limited applications, especially on stress areas, which is attributed to their inferior mechanical properties.^(14,17, 18) New types of GICs have been introduced by the addition of resins, such as HEMA, to the conventional GIC formulation. The so-called resin-modified GIC (RMGIC) relatively possesses the same shortcomings.^(20,21) Several aspects have been studied in order to improve the mechanical and physical properties of GICs, such as composition⁽¹²⁾, viscoelastic properties⁽²²⁾, fracture toughness^(16,22),

powder particle size reduction⁽¹⁶⁾ radio-density⁽²³⁻²⁵⁾, strength^(10,15) and powder/liquid (P/L) ratio.⁽²³⁾ The diametral tensile strength (DTS) is one of the most common and useful mechanical properties of GICs, being extensively found in many studies.^(12,17, 22,26) Depending on the clinical application, GICs should be strong enough to resist stresses generated within it when loaded with occlusal forces, as it is truth for luting materials⁽¹⁰⁾. The type of material, filler size and proportion, addition of metals, composition and P/L ratio are factors, which affect strength of GICs^(10,16,17,12,22,27). Increased P/L ratios tend to increase DTS of GICs.⁽¹⁰⁾

True that powder/liquid (P/L) ratio is one of indicative factors in altering mechanical properties of GICs; the higher the amount of powder, the higher the mechanical properties.^(20,28,29) This correlation has been explained through the particles of the powder that remain unchanged due to lower level of acid. It has been proposed that these unreacted particles appear like filler in such a way that they prevent crack propagation within the cement.⁽²⁰⁾ The powder amount of the cement has a direct correlation with compressive strength, porosity and wear resistance and an indirect correlation with solubility, setting and working time.⁽³⁰⁻³¹⁾ Accordingly, this is obvious that P/L ratio can significantly affect the mechanical properties of GICs and their clinical performance as well. The P/L ratio for clinical use may vary from one manufacturer to another because of several factors.⁽³⁶⁾ Visual scaling and careless use of scaling scoop can affect this ratio adversely.^(28,31,32,33) The liquid drop can also vary in volume due to bottle inclination during dispensing, shape of the bottle outlet, as well as presence and size of air bubble in the bottle.^(28,32) Nevertheless, even an accurate use of the scoop and bottle leads to 2-8% of variation in P/L ratio.⁽³⁴⁾ Variation in P/L ratio (approximately 6%) in capsulated GICs has also been reported.⁽³⁵⁾ Consequently, the aim of this study was to evaluate the effect of different P/L ratios on the tensile and shear bond strength to dentin.

MATERIALS AND METHOD

Samples preparation for tensile strength test

An upper right maxillary first molar was mounted in a 10x10x20 mm stone block. The molar was prepared to a 0.5 mm chamfer finish line using round-end taper diamond bur with guiding pin # 8881 P (Komet, Brasseler, Germany). 1.5 mm occlusal reduction was done using round-edge wheel stone (5909-314-040, Komet, Brasseler, Germany). Using industrial silicon (Body Double, Smooth-On, Inc., Pennsylvania, USA), a mold was fabricated for the stone block. 60 replicas for were fabricated using acrylic resin (Vertex-Dental B.V., The Netherlands). A wax coping was fabricated on the stone block. A silicon index was fabricated to the wax coping, then filled with molten wax and mounted on each resin die to construct 60 standardized wax copings. The margins were then reflowed and adjusted using marginal wax (Renfert GmbH, Hilzingen, Germany) to ensure proper marginal integrity. An occlusal loop was attached to each coping. Wax patterns were sprued and invested each 12-17 copings in one ring to be cast into Co-Cr copings (Remanium Star, Dentaureum, Germany). Copings were finished and inspected for any deformity. They were air particle abraded using 100 μ m Al₂O₃ at 80 psi and then were cleaned using ultrasonic water bath. Copings were labeled and

divided into 4 groups according to P/L ratio used. (Table 1) GIC cement was manipulated according to manufacturer's directions and applied to the fitting surface of metal coping. Each coping was cemented to its corresponding resin die and held under static load of 5 kg. for 10 minutes. Excess cement was removed using a sickle scaler. (Fig.1 A)

TABLE (1) Sample grouping

Group	Powder/Liquid Ratio	No. of samples
A	1 scoop/1 drop	15
B	1 scoop/2 drops (control)	15
C	1.5 scoops/1 drop	15
D	2 scoops/1 drop	15
Total		60

Samples preparation for Shear bond strength test

Twenty-Eight caries and restoration-free human upper right maxillary first molar of approximate sizes were collected. Teeth were cleaned and sterilized in an autoclave at 121°C, 15 Psi for 40 minutes. Teeth were then embedded completely in an auto-polymerized transparent acrylic resin (Vertex-Dental B.V., The Netherlands) blocks with a size of 10 x 10 x 20 mm. After complete setting, the blocks were mounted and trimmed on a trimmer to expose 4x4 mm patch of dentine on their buccal side. The exposed dentine surface was cleaned,

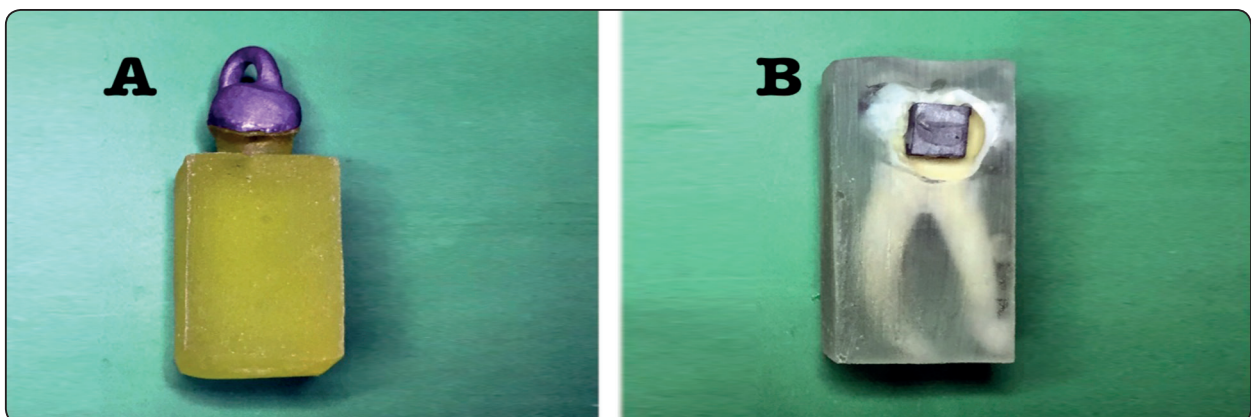


FIG. (1) A: Cemented copings over resin dies. B: Cemented Cr-Co cube to exposed dentine surface.

polished using polishing paste on a rotary cup. A wax cube was constructed having dimensions of 3x3 mm and thickness of 2 mm. A silicon mold was constructed for standardized fabrication of 60 wax pattern cubes. The wax cubes were cast into Cr-Co cubes, sandblasted, ultrasonically cleaned and dried. GIC cement was manipulated according to manufacturer's directions and applied to the fitting surface of metal cubes. Using a tweezers each cube was cemented to its corresponding patch of dentine and held under static load of 5 kg. for 10 minutes. Excess cement was All Tensile and shear bond strengths specimens were stored in distilled water for 72 hours, then thermocycled for 300 cycles with the sequence of 20 sec at 5°C and 20 sec at 55°C and 10 sec transport. (Fig.1 B)

Tensile strength testing

Samples were submitted to a tensile test using a universal testing machine (Instron 8874; Instron Corp. Canton, Mass) under a constant crosshead speed of 1.00 mm/min. The fracture was confirmed by sudden drop in force measurements in the testing machine. (Fig.2)

Shear bond strength testing

A square interface shear test was designed to evaluate the bond strength. All samples were individually mounted on a computer-controlled materials testing machine (Model LRX-plus; Lloyd

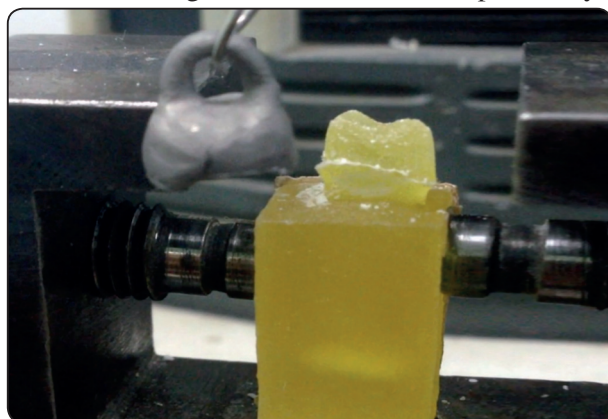


Fig. (2) A Sample during tensile testing.

Instruments Ltd., Fareham, UK) with a loadcell of 5 kN and data were recorded using computer software (Nexygen-MT; Lloyd Instruments). Samples were secured to the lower fixed compartment of testing machine by tightening screws. Shearing test was done by compressive load applied at Cr-Co dentine interface using a mono-bevelled chisel shaped metallic rod attached to the upper movable compartment of testing machine traveling at cross-head speed of 0.5 mm/min. The load required to debonding was recorded in Newton.

Bond strength were calculated using the following equation

The load at failure was divided by bonding area to express the bond strength in MPa : $\tau = P/L^2$ where; τ =shear bond strength (MPa, P =load at failure(N) and L =arm of the square(mm).

RESULTS

The mean and standard deviation of tensile force values between groups, are summarized in. (Table 2 & Chart 1) The highest mean value was observed in group C (409.25 MPa) and the lowest mean value was observed for group A (235.69 MPa). Statistical analysis revealed significant difference between groups ($P= 0.010$). Statistical analysis among groups showed significant difference between groups A and C ($P= 0.017$), between groups B and C ($P= 0.003$) and between groups B and D ($P= 0.033$). But no significant differences were found between groups A and B ($P= 0.879$), groups A and D ($P=0.062$) or between groups C and D ($P= 0.731$). (Table 3) Regarding shear bond strength, the mean and standard deviation values between groups, are summarized in Table 4 & Chart 2. The highest mean value was observed in group A (8.39MPa) and the lowest mean value was observed for group B (1.06 MPa). Statistical analysis revealed significant difference between groups ($P= .000$). Statistical analysis among groups showed significant difference between groups A and B ($P= 0.000$), between groups

A and C ($P=0.002$), between groups A and D ($P=0.001$) and between groups B and D ($P=0.032$). But no significant differences were found between groups B and C ($P=0.063$), or between groups C and D ($P=0.870$). (Table 5)

TABLE (2) Mean and standard deviation of tensile force values of the tested groups.

P/L	Mean	Standard deviation
Group A	235.69	113.91
Group B	244.76	61.12
Group C	409.25	60.54
Group D	389.26	109.95

TABLE (3) One-way ANOVA tensile force values among tested groups.

	Group A	Group B	Group C	Group D
Group A		0.879	0.017	0.062
Group B	0.879		0.003	0.033
Group C	0.017	0.003		0.731
Group D	0.062	0.033	0.731	

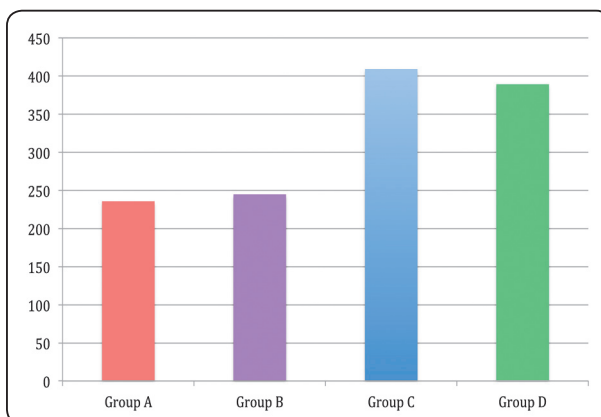


CHART 1: Mean tensile force values of tested groups

TABLE (4) Mean and standard deviation shear bond strength values of the tested groups.

P/L	Mean	Standard deviation
Group A	8.39	1.75
Group B	1.06	1.04
Group C	3.20	1.96
Group D	3.02	1.34

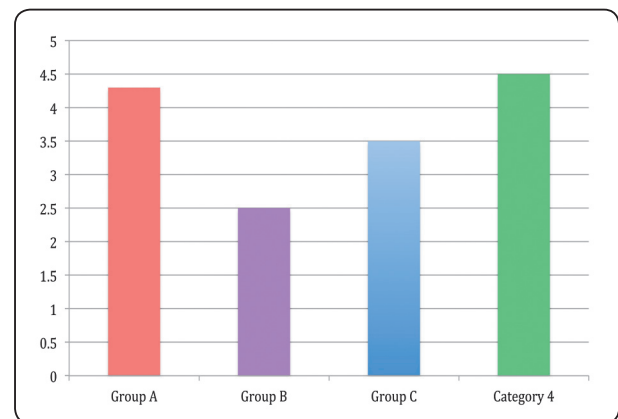


CHART 2: Mean shear bond strength values of tested groups.

TABLE (5) One-way ANOVA shear bond strength values among tested groups.

	Group A	Group B	Group C	Group D
Group A		0.000	0.002	0.001
Group B	0.000		0.063	0.032
Group C	0.002	0.063		0.870
Group D	0.001	0.032	0.870	

DISCUSSION

The correct choice of a luting agent is key to success in indirect dentistry and dependent on the clinical situation, based on its physical, biological and handling properties.⁽³⁷⁾ Changes in the powder-liquid ratio of conventional glass ionomer cements may alter some physical properties. One

of them is the film thickness, which depends on multiple factors⁽³⁸⁻⁴²⁾ including manipulation, temperature of the mix, consistency, handling, working and setting time and time elapsed after mixing.^(38,40,41,43-45) Consistency is considered one of the principal features affecting the film thickness of any luting material.⁽⁴¹⁾

Variation in the P/L ratio is an important factor in optimizing GIC properties. Nevertheless, the amount of powder used under clinical conditions is often overlooked. Billington et al.⁽⁴⁶⁾ (1990) showed that this amount was 27% less than that recommended by the manufacturer. This finding was confirmed by Beher et al.⁽⁴⁷⁾ (2006) who pointed out a 17% variation range. In addition, some studies showed that the more the amount of powder, the better the mechanical and physical properties⁽⁴⁷⁻⁴⁹⁾ and have suggested to use the most possible powder amount the strength of GIC is required.⁽⁵⁰⁾

The results of this study indicated that the increase in the P/L ratio improved tensile and shear bond strength. This is in agreement with other researchers that concluded that variation of the P/L ratio increased mechanical properties of GIC.^(9, 20, 31, 49, 51,52) On the other hand few reaches found that variation in the P/L ratio other than manufacturer direction had no significant difference in bond and flexural strengths or mechanical properties.^(36,53)

It's quite important to mention that in group C (1.5 scoops/1 drop), the mixing was more difficult than the control group and needed speed and skill. Moreover the mixing on group D was even more difficult and needed speed and skill in placing both the cement in the coping and in seating the coping. The manufacturer recommendation (group B) although gave inferior mechanical properties to other groups but indeed was the easiest in mixing and handling.

As mentioned before, some researchers believe that greater powder amount can improve the

mechanical properties due to the filler-like function of unreacted powder particles.⁽²⁰⁾ In fact, in a recent study, it was shown that the increase in P/L ratio of a RMGIC luting cement by 30 percent resulted in an increase in compressive, flexural and bond strengths.⁽⁵⁴⁾

However, no increase was observed when P/L ratio increased by only 20%.⁽³⁶⁾ In addition, Fonseca et al.⁽⁵⁵⁾ (2010) found that a 50% alteration in P/L ratio produced a weaker RMGIC in diametral tensile strength.

True that, the type of material, the investigated property and also the amount of unreacted particles are critical factors to be considered. These factors may explain the concordance between the present study and that of Fleming et al.⁽³⁰⁾ (2003), in which the compressive strength of Chemfil was investigated and significant differences were achieved.⁽³⁰⁾ Lund et al.⁽⁵⁶⁾ (2007) It was also reported that the effect of P/L ratio on the diametral tensile strength of RMGIC is brand dependent. However, it is noteworthy that diametral test is only recommended for brittle materials.

Due to the vast disparity of P/L ratios and the results reported, it is very challenging to make a definite conclusion concerning the ideal P/L ratio. For instance, Mitsuhashi et al.⁽⁴⁸⁾ (2003) found no significant difference in fracture strength and mechanical properties of RMGICs by reducing the amount of powder by 33%. Furthermore, a recent study indicated no significant difference in flexural strength of RMGIC after 17 and 25% decrease and 33 and 50% increase in powder level.⁽⁴⁷⁾

The majority of the previous studies have considered the reduction in powder amount^(19,48,49). Problems attributed to the incorporation of high powder content during mixing procedure have encouraged investigations on the decreased P/L ratio.⁽⁴⁸⁾

This study also evaluated the effect of P/L ratio on the bonding strength; this has not been examined

before, which can be due to difficult specimen preparation.⁽³¹⁾

In the present study, mixing the cement with greater powder amount was difficult, but the handling was improved due to less adhesiveness, that immediately lost homogeneity and made the application difficult and almost impossible.

Under clinical conditions, as the increase in powder amount did not show adverse effects on mechanical properties, it can be recommended to increase the powder amount to achieve better handling properties. In this case, the clinical longevity of GIC restorations will probably increase.

CONCLUSIONS

Within the limitations of the study it could be concluded that,

- There were significant difference between groups in both the shear bond strength and the tensile strength.
- The increase in the P/L ratio to 1.5 scoops/1 drop or 2 scoops/1 drop improved tensile and shear bond strengths Of hand mixed GIC than the manufacturer's recommendation.
- The 1 scoop/1 drop group improved shear bond strength but not tensile strength of hand mixed GIC than the manufacturer's recommendation.

Further researches are needed to confirm this finding along with film thickness and marginal integrity.

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