Variation of powder/liquid ratios of capsulated glass-ionomer materials

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Abstract

Background and objective: Besides hand-mixed formulations, glass-ionomer restorative materials are also available in sealed capsules. The main advantage of capsulated materials is that clinicians cannot alter the powder/liquid ratio. This research was conducted to investigate the consistency of the recommended powder/liquid ratio by the manufacturer in relation to the actual content of the capsule and to determine whether these variations from the manufacturers' recommendations have any influence on the surface microhardness and compressive strength.

Methods: Two different batch numbers for five glass-ionomer restorative materials and one Carbomer were evaluated. Twenty specimens were selected from each batch of materials: Chemfil Rock (Dentsply Sirona), Fuji Equa Forte (GC Corp), Glass Fill (GCP dental), Ionofil Molar AC (Voco) and Ketac Universal (3M ESPE) and Riva Self Cure (SDI Limited). The capsules were deconstructed and the powder and liquid were weighed. Additionally, five specimens were prepared for each time interval (days 1, 7 and 180) and the compressive strength assessed. The surface microhardness (Vickers hardness) was completed on five specimens per batch and assessed sequentially at days 1, 7 and 180 on the same specimen after the elapsed time intervals.

Results: The results showed that there were significant differences between the two batches for the powder and liquid content for Chemfil Rock (p<0.001), Ketac Universal (p=0.029) and Riva Self Cure (p<0.001). There were no significant differences for surface micro-hardness or compressive strength between the two batches for any of the assessed time intervals (days 1, 7 and 180).

Conclusion: There were differences between the various batches from the same manufacturer concerning the powder/liquid ratio content. The actual content of powder and liquid from the two batches differed when compared to the manufacturers' recommended powder/liquid ratio on the package insert for most of the manufacturers. The difference in powder/liquid ratios between batches did not significantly influence the Vickers hardness or the compressive strength for the material over the same time period.

Keywords: Capsulated, Glass-ionomer, Carbomer, powder/liquid ratio, Vickers hardness, compressive strength, powder weight, liquid weight.

Abbreviations and acronyms:

CR	=	Chemfil Rock
Batch	=	B
FEF	=	Fuji Equa Forte Fil
GF	=	GCP Glass Fill
GICs	=	Glass ionomer restorative materials
IM	=	Ionofil Molar AC
KU	=	Ketac Universal
PLR	=	Powder/liquid ratio
RSC	=	Riva Self Cure
RMGIC	=	Resin modified glass-ionomer

Introduction

The favourable bioactive properties of glass-ionomer restorative materials (GICs) lie in the ability to form a physicochemical bond to the tooth structure (Glasspoole et al., 2002). This physicochemical bond is primarily due to ion exchange and integration to moist tooth structure (Wilson et al., 1983; Yoshida et al., 2000). GICs also have a thermal compatibility, since the coefficient of thermal expansion is similar to that of dentine (Nassan and Watson, 1998). The anti-cariogenic properties due to a release of fluoride (Xie et al., 2000; Chau et al., 2015) and other ions are advantageous to both sound tooth structure and compromised tooth structure (Ngo, 2005). GICs can be technique sensitive and two key disadvantages of GICs may include low early strength and moisture sensitivity during the initial setting process (Pelka et al., 1996; Xie et al., 2000). The setting process of conventional GICs are characterised by an acid-base reaction between the powder and the liquid. Powder/liquid ratios (PLR) were therefore shown to play an important role in the early stages of material placement and maturation (Eames et al., 1977). Initially, when GICs were launched, the method of mixing was by spatulation and capsulated versions were readily available. The clinical manipulation by clinicians is based on two main variables namely: 1) variation in the volume of incorporated powder and 2) the speed of spatulation has been well documented for hand-mixed materials (Billington et al., 1990; Cattani-Lorente et al., 1993). The challenge that clinicians face with hand spatulation of GICs in daily dental practice is to abide by the manufacturer's mixing time and PLR instructions.

The capsulated materials should have an advantage over hand-mixed preparations as the manufacturer controls the PLR of the components. Hand-mixed materials are vulnerable to variability of mixing and

PLR (Nomoto, 2004; Mulder, 2018). Mechanical mixing together with the pre-proportioned PLR of capsulated GICs by the manufacturer should therefore allow the various functional properties of the mixed material to be less susceptible to operator-induced variability. The use of capsulated GICs is considered to be less time consuming, as it is ready for immediate use by expressing the GICs into the preparation directly from the capsule and a cleaner clinical area is maintained than when hand-mixed GICs are used (Nomoto and McCabe, 2001). With capsulated materials, mixing speed (Gee and Pearson, 1993) and duration of mixing (Fleming et al., 2006) can be influenced by the clinician. Manipulation of the mixing speed and duration of the GICs in capsulated form can either increase or decrease the setting time. This could however result in a decreased PLR, especially if the mixing speed is lower than 3000 oscillations (Rupp et al., 1996). The decreased PLR is due to the powder not achieving a sufficient mix with the liquid. As a result, the non-reacted powder decreases the PLR that can take part in the acid-base reaction. The alteration of the PLR can result in the GICs having insufficient liquid for the wetting and interaction with the powder. The polyalkenoic acid portion of the liquid interacts with the powder to allow various metallic ions to release from the glass particles to form the primary polysalt matrix (Maeda et al., 1999). Subsequent to metallic ion release a siliceous hydrogel is left on the glass particle surface (Hatton and Brook, 1992; Nicholson, 1998). Therefore, the interactions of the liquid with the powder influence the amount of ions released and directly influence the matrix formation. Although the GICs continue to absorb water into the GICs from the moist dentine, it allows additional acid-base reactions to take place over time (ten Bosch *et al.* 2000). The absorbed moisture is essential for the gradual maturation and increase of compressive strength due to the hydration of the silicate phase that contribute to the increase of strength (Matsuya et al., 1996). In order to assess the effect of maturation and the variations of the capsulated PLR of the GICs, the time lapse was assessed with specimens matured in deionised water. It has been shown that GICs mature over time and this changes their physical properties (Zanata et al., 2011; Zoergiebel and Ilie, 2013).

The bond strength of GICs to both healthy and carious tooth structure has been studied with shear bond testing (McInnes-Ledoux et al., 1989; Burke and Lynch, 1994). The use of GIC restorations for the ART technique have been shown to be clinically successful in single surface restorations after a three year period (Frenken et al., 1998; Holmgren et al., 2000), although the bond strengths of GICs have been reported to be in the range of 3±4 MPa (Hewlett et al., 1991; Ewoldsen et al., 1997). The more modern high viscosity glass-ionomers assessed in this study have shown much better physical properties and Ketac Universal (3M) and Fuji Equa Forte Fill (GC) have expanded indications for restorative treatment. The manufacturer classifies Glass Fill as a Glass Carbomer due to the filler particles in the material. The claimed improvement in the physical properties of the Carbomer is claimed by the manufacturer to be

attributed to the nano sized apatite filler particles. The material is advocated to be thermocured with a LED curing light through the surface protective gloss (Arita et al., 2011).

The accuracy of the PLR in GICs has been questioned. A study using three GICs, with a PLR of -11%, +3% and +6% of the manufacturer's recommendations respectively, was identified (Gee and Pearson, 1993). Variations in PLR of ~6% have been reported in capsulated materials (Azillah et al., 1998).

The rationale for this present study was that as GICs mature over time, it was uncertain how the possible variation in PLR would influence it. Time intervals of assessment for the Vickers hardness and compressive strength were completed on days 1, 7, and 180. Ionofil Molar AC (Voco) was only assessed for powder/liquid ratios, due to insufficient capsules per batch.

The purpose of the study was to assess the consistency of the PLR in capsulated GICs and a Carbomer in relation to the manufacturer's recommendations. It was important to assess two different batch numbers from each manufacturer and compare them to recommended capsulated content. This allowed insight to any differences in the batches from each manufacturer compared to the PLR manufacturer recommendation as stated on the packaging. The subsequent research question was whether a variation in the PLR between batches and/or the manufacturers' recommendations would influence the Vickers hardness and compressive strength. As the material matures, the Vickers hardness and compressive strength change, moisture is absorbed and ions are released (De Caluwe et al., 2017).

Materials and Methods

Figure 1 provides an outline of the capsule distribution for the powder/liquid assessment and comparison of the compressive strength and surface microhardness of the two batches of materials. IM could not be assessed for Vickers hardness nor compressive strength due to insufficient capsules per batch.

Powder/liquid assessment and capsule deconstruction
Two different batches (B1, B2) from each manufacturer were assessed. Five materials of encapsulated GICs and one Carbomer were assessed for their powder and liquid content (Figure 2, 3). Twenty capsules were randomly selected from each of the two different batch numbers (n=240) for the PLR assessment (Table 1). The capsules were deconstructed in order to access the liquid weight followed by the powder weight. The techniques used to deconstruct the capsules were based on the capsule design. Figure 2 shows the posterior plunger system for Chemfil Rock (CR), Fuji Equa Forte (FEF), Riva Self Cure (RSC) and the Carbomer namely GCP Glass Fill (GF). The remaining GICs namely Ketac Universal (KU) and Voco Ionofil Molar AC (IM) had the liquid in a compressible diaphragm located on the superior aspect of the capsule under the activation cover (Figure 3).

Capsule deconstruction of CR, FEF, GF, and RSC (n=160): The capsules were stored with the dispensing tips down for 30 minutes prior to capsule deconstruction.
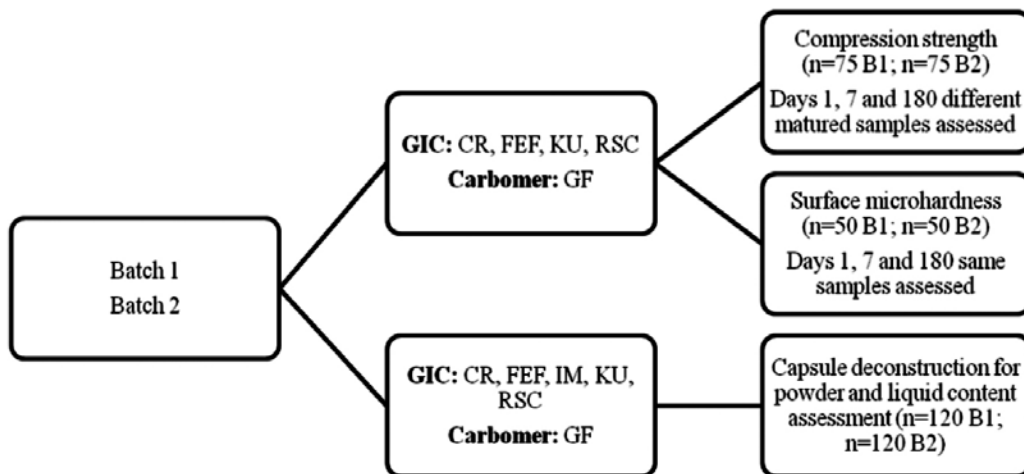
Figure 1. Flowchart of capsule distribution for specimen preparations

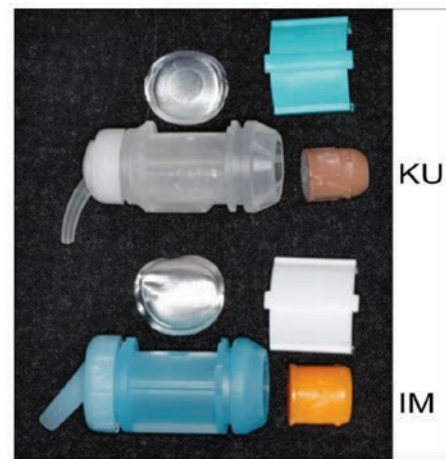**Figure 2.** Capsule systems with a posterior plunger.

Figure 3. Capsule systems with a diaphragm system.

Table 1. Material abbreviation, batch numbers and manufacturer PLR recommendation.

Material and manufacturer	Material abbreviation	Recommended powder (g)	Manufacturer recommended liquid (g)	Manufacturer recommended PLR
CR : Chemfil Rock (Dentsply Sirona, Konstanz, Germany). B1: 1511000724 B2: 1502008003	CR B1 CR B2	0.442	0.12	3.683 : 1
FEF : Fuji Equa Forte (GC Corp, Tokyo, Japan). B1: 1508265 B2: 151118A	FEF B1 FEF B2	0.4	0.13	3.076 : 1
GF : Glass Fill (GCP dental, Netherlands). B1:7511350 B2:75114444	GF B1 GF B2	0.5	0.15	3.333 : 1
KU : Ketac Universal (3M ESPE, Seefeld, Germany). B1: 614726 B2: 606207	KU B1 KU B2	0.339	0.106	3.198 : 1
RSC : Riva Self Cure (SDI Limited, Australia). B1: B1506291F B2: B1508193F	RSC B1 RSC B2	0.45	0.14	3.214 : 1
IM : Ionofil Molar AC (Voco, Germany). B1: 1530546 B2: 1526687	IM B1 IM B2	0.43	0.125	3.440 : 1

This storage position allowed the dispensable liquid in the liquid compartment to pool at the bottom of the liquid chamber. The plunger (Figure 2) was removed carefully with pliers. Liquid present on the plunger was wiped off with one of three discs of filter paper, to ensure that no liquid adhered to the plunger due to surface tension. The liquid was absorbed from the chamber with three identical discs of Whitman filter paper (Whitman no1, GE Healthcare, Wood Dale, IL, USA) which was punched with a 25 mm paper punch (Upikit HCP-110 craft punch, circle shape size one, Upikit International PTY Ltd, Tainan, Taiwan). The three filter paper discs of known weight were held with a stainless steel tweezer and the liquid absorbed from the liquid chamber. Visual inspection under 1.75x fluorescent magnification (Start International, Dallas, TX, USA) ensured that no liquid remained in the chamber. The liquid content was calculated by deducting the known weight of the three filter paper discs from the final weight after the liquid was absorbed. The chamber that housed the liquid was subsequently removed with a carbon surgical blade (Swann-Morton, Sheffield, England) to expose the powder chamber. The powder was placed in a small specimen jar of known weight. A visual inspection under magnification as previously described was performed to ensure that no powder was retained on the capsule walls.

KU, IM (n=80): These capsules were not stored with the dispensing tip down for 30 minutes, since the liquid component was pre-packed in a silver compressible diaphragm on the superior aspect of the capsule under the activation cover (Figure 3). The various plungers were removed carefully with pliers to gain access to the powder chamber. The powder was placed in a small specimen jar of known weight. Visual inspection under magnification as previously stated ensured that no powder was retained on the capsule walls. Subsequently the silver compressible diaphragm was weighed with the liquid content. The dispensable liquid was determined by using a surgical blade (Swann-Morton no.15) to cut the diaphragm and weighing the liquid on the three filter paper discs. The silver diaphragm was visually inspected as previously described. The liquid weight was additionally confirmed by deducting the initial weight of the diaphragm from the end weight of the empty diaphragm.

After deconstruction, the weight of the powder and the liquid were established on a desktop analytical balance (Ohaus Precision Standard, Model TS400D, Ohaus Corp, Florham Park, N.J, USA). The PLR was calculated by dividing the powder weight by the liquid weight and comparing it to the manufacturer's recommendation (Table 1).

Compressive strength

Four GICs (except IM) and one Carbomer were assessed for compressive strength (Figures 2 and 3). Five cylindrical test specimens were prepared for each material and batch for the various time intervals (days 1, 7 and 180). The specimens were prepared and assessed as per the ISO for Dentistry-Water-based cements– Part 1: Powder/liquid acid-base cements (ISO 9917-1).

For the mixing of the materials, the ISO 9917-1 standard indicated a relative humidity of 50 ± 10%. The mould and clamp used to prepare the specimens were kept in a temperature-controlled cabinet (37°±1) with a 30% relative humidity. The capsules were mixed with a Rotomix (3M ESPE, Seefeld, Germany) according to the manufacturers' instructions, except for GF. The only deviation was where GF did not receive the surface protective gloss followed by thermocuring with a LED-curing unit through the gloss, directly after material placement in the mould. This was omitted, since the mould and clamp set-up outlined in ISO 9917-1 was not conducive to those two steps. All the specimens were prepared at room temperature (23°±1). The cylindrical test specimens were made by placing the mixed materials into a Teflon split mould and stainless steel plates (height 6±0.1 mm, diameter 4±0.1 mm). The mould was slightly overfilled and a cellulose strip was placed between the material and the plates. The specimens were removed from the moulds after one hour of bench setting and immediately submerged in 5 mL deionized water grade 3 (37°C ±1), as defined in ISO 3696:1987. The specimens were stored in this medium for 1, 7 and 180 days in a temperature-controlled cabinet. The compressive force (p) was measured in Newton with a Universal Testing Machine (H10KT-0293 Tinus Olsen, Redhill, England) at a cross-head speed of 0.75 mm/min. The tester software was used for analysis of the results (QMat Testzone, version 4.5.37, Tinus Olsen). The specimens (with moist filter paper on either side) were placed with their flat ends between the plates on the testing machine, so that the progressively increasing compressive load was applied along the 6 mm long axis of the specimen.

The compressive strength was calculated in megapascals (MPa), using the following equation:

$$C = 4p/\pi d^2$$

C: Compressive strength in MPa
p: Compressive force/maximum force applied, in Newton
d: Diameter of the specimen, in millimeters
π: Constant for pie used as 3.14

Surface microhardness analysis

The surface microhardness of four GICs (except IM) and one Carbomer (Figure 2, 3) were assessed with a Vickers hardness indenter. Five specimens were constructed and assessed at the various time intervals (days 1, 7 and 180).

Storage was completed as described for the specimens for compressive strength. Forty-eight hours after the specimens were submerged in distilled water, the surface of all the specimens for Vickers hardness testing were smoothened with 2500 grit silicon carbide paper, followed with 4000 grit (3M, Maplewood, MN, USA). The polishing ensured an adjustment to the surface of ±100 µm (as measured within a micrometer). The application of the carbide paper simulates the restoration polishing that would occur in the clinical setting (Menne-Happ and Ilie, 2013). This smoothed surface allowed

accurate evaluation of the pyramidal indentation on the surface of the material. A second and blinded operator completed the surface hardness evaluation with a Vickers hardness indenter (Zwick-Roell durometer, ZHV1/2 Micro-vickers, Italy) set at HV0.5 (load of 500 gf) and a dwell (indentation) time of ten seconds. The instrument automatically calculated the Vickers hardness after the diagonals were selected. Three Vickers hardness (VH) measurements were recorded 500 μm apart from one another on each specimen to obtain a mean value per specimen. The mean values from the five specimens of each batch per time period (days 1, 7 and 180) were calculated with a standard deviation and compared per batch within the same material group (Ellakuria et al., 2003; Yap et al., 2003; O'Brein et al., 2010).

Statistical analysis
Data were tabulated in a Microsoft Excel® spreadsheet (2012); (Microsoft® Corp., Richmond, VA, USA) and analysed. The statistical analysis included the calculation of p-values at a significance level of p<0.05 for all analyses. The powder/liquid ratio was assessed using

the Bartlett's test for homogeneity of variances in the standard deviation (SD). The mean values of B1 and B2 for each material was analysed with the Welch version of the two-sample t-tests, which took the differences in the variances into account to establish the significance of the differences. For the Vickers hardness and compression test, the Welch t-test was completed to establish if there was a significant difference between the two batches. In order to consider the changes in the physical properties as the GICs and the Carbomer mature, the difference in the two batches for Vickers hardness and compressive strength were assessed at days 1, 7 and 180.

Results
Powder/liquid assessment in the capsules
The statistical analysis between B1 and B2 within the material groups are represented in Table 2 for the mean PLR and SD of the twenty capsules from each batch number. Significant differences were found between the mean values of the PLR from the 20 specimens from B1 and B2 within the material for CR (p<0.001), KU (p=0.029) and RSC (p<0.001). The variances of the observed PLR inside the capsules were assessed in terms of the SD for the batches. The Barlett's test indicated significant differences between the SD B1 and SD B2 for GF (p=0.012), IM (p=0.011) and KU (p=0.004). GF B1, IM B2 and KU B2 had the largest SD within their respective manufacturer batches, resulting in the significant difference between the batches within the manufacturer. This variance can be seen in the spread within each batch between the 20 PLR observations represented in Figure 4.

Differences were present between batches shown in Table 3 based on the PLR mean values of the observation. Table 3 presents the smallest difference between

Table 2. Powder liquid ratio mean value of batches.

Material	Mean PLR B1	Mean PLR B2	p–value of mean
CR	3.4712(±0.05767)	3.6238(±0.07146)	<0.001
FEF	3.4483(±0.08895)	3.4336(±0.09109)	0.607
GF	3.5665(±0.14918)	3.5062(±0.08779)	0.129
KU	3.2145(±0.04234)	3.1680(±0.08004)	0.029
RSC	3.4788(±0.04625)	3.2368(±0.05712)	<0.001
IM	3.6068(±0.03131)	3.5849(±0.05384)	0.126

Table 3. PLR comparative difference between batches and the manufacturer recommendation.

Material	Mean powder in capsule	% Powder difference between batch and manufacturer recommendation	Mean liquid in capsule	% Liquid difference between batch and manufacturer recommendation	Mean PLR capsules	PLR % difference between B1 and B2	PLR % difference between batch and manufacturer recommendation
CR B1	0.4516	+2.17	0.1301	+8.41	3.4712 : 1	4.39	-5.75*
CR B2	0.4569	+3.37	0.1261	+5.08	3.6238 : 1		-1.60*
FEF B1	0.3986	-0.35	0.1156	-11.07	3.4483 : 1	0.42	+10.79
FEF B2	0.3928	-1.8	0.1144	-12	3.4336 : 1		+10.41
GF B1	0.4115	-17.7	0.1151	-23.06	3.5665 : 1	1.69	+6.63
GF B2	0.4123	-17.54	0.1176	-21.60	3.5062 : 1		+5.02
KU B1	0.3384	-0.17	0.1053	-0.66	3.2145 : 1	1.48	+0.52*
KU B2	0.3339	-1.50	0.1051	-0.56	3.1680 : 1		-0.94*
RSC B1	0.4129	-8.24	0.1187	-15.21	3.4788 : 1	6.95	+7.61*
RSC B2	0.4143	-7.93	0.1280	-8.57	3.2368 : 1		+0.63*
IM B1	0.4256	-1.02	0.1180	-5.6	3.6068 : 1	0.60	+4.62
IM B2	0.4240	-1.39	0.1183	-5.36	3.5849 : 1		+4.04

+ indicate more than manufacturer recommendation. - indicate less than manufacturer recommendation.
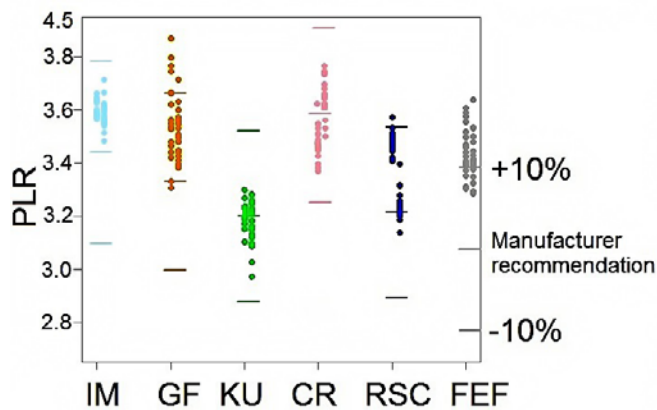* significant difference between the two batches from the same manufacturer.

Figure 4. Powder/liquid ratio distribution of two batches.

Table 4. Variation of the capsules' PLR distribution from the manufacturer recommendation.

Material	Number of capsules with a PLR -10% to 0%	Number of capsules with a PLR 0% to 10%	Number of capsules with a PLR >10%
IM B1	0	20	0
IM B2	0	20	0
GF B1	0	15	5
GF B2	0	19	1
KU B1	5	15	0
KU B2	10	10	0
CR B1	20	0	0
CR B2	16	4	0
RSC B1	0	19	1
RSC B2	0	20	0
FEF B1	0	4	16
FEF B2	0	5	15

0% represents the exact manufacturer recommended PLR

the two batches from the same manufacturer were FEF (0.42%) followed by IM (0.60%). The greatest differences between the actual mean PLR per batch and the manufacturers' recommendation on the side of the packaging was FEF (B1:10.79%. B2:10.41%). The 240 PLR observations were plotted for each manufacturer; B1 on the left and B2 on the right (Figure 4). The upper (10%) and lower horizontal bars (-10%) represent the variation of the PLR from the manufacturers' instructions that ensure no significant change to the setting time, compression strength or flexural strength. The actual manufacturers PLR recommendation has been placed as the horizontal bar between the -10 to 10% bars (Figure 4, Table 4).

Table 4 additionally illustrates the distribution of the observations located between the -10% to 10% PLR from the recommended manufacturer PLR. The batches

from CR B1, CR B2, KU B1, KU B2 and RSC B2 had observations that were well distributed around the PLR of the respective manufacturer's recommendation. IM, CR, KU and RSC B2 were the only material batches with all 20 PLR observations between the -10% to 10% range from the manufacturers' recommended PLR. (Figure 4, Table 4). This does however not illustrate that the silver diaphragm is superior in terms of liquid accuracy compared to a liquid chamber. When Tables 1 and 3 are interrogated on how the mean values of powder and/or liquid can vary individually from the manufacturer's recommendation, it becomes clear that the PLR as a stand alone value must not be misinterpreted, as the final PLR is close to the ratio recommended by the manufacturer despite the individual variation from the manufacturer individual powder and liquid values. Therefore, CR B1, CR B2, KU B1 and KU B2 were the material manufacturers that displayed individual powder, individual liquid and combined PLR closest to the manufacturer recommendation. The spread of the observations that were significantly different can be considered irrelevant for IM and KU, since the observations are all within the -10% to 10% PLR from that of the manufacturer.

Surface microhardness
The Vickers hardness values between the batches of the same material and the same specimens were compared for each time interval to assess how it differed as the material matured (Figure 5). The hardness for the materials with the exception of RSC B1 day 7 showed an increase in surface hardness over the progression of days 1, 7 and 180. The statistical analysis revealed no significant differences for each manufacturer between Batch 1 and Batch 2 for any of the materials tested within the same time period of days 1, 7 and 180 ($p > 0.05$).

Compressive strength
The compression strength values between the batches of the same material, but different specimens for days 1, 7 and 180 were compared (Figure 6). All the materials indicated an increase in compressive strength over the progression of days 1, 7 and 180 with the exception of KU B1 day 7, CR B1 day 7, CR B2 day 7, RSC B1 day 7 and RSC B2 day 7, with no clear explanation as the specimens were all produced at the same point in time prior to storage. These specimens interestingly all had a lower mean compressive strength on day 7 compared to the comparative compressive strength values at days 1 and 180. The aim of study was however still achieved since this decrease on day 7 for these batches did not show any statistical differences between the batches for each manufacturer.

Discussion
The present study rejected the hypothesis that the PLR will be similar between batches for CR, KU and RSC. Further, the hypothesis of the variance in the PLR in the capsules between the batches will not differ for GF was rejected. For IM and KU, the variances will not differ significantly was accepted, since all the observations
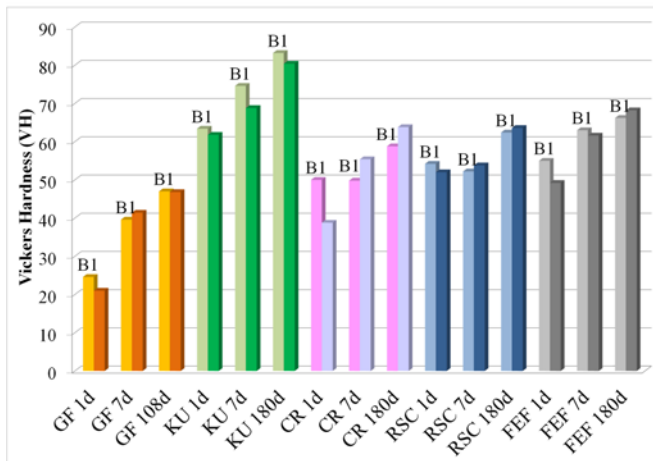
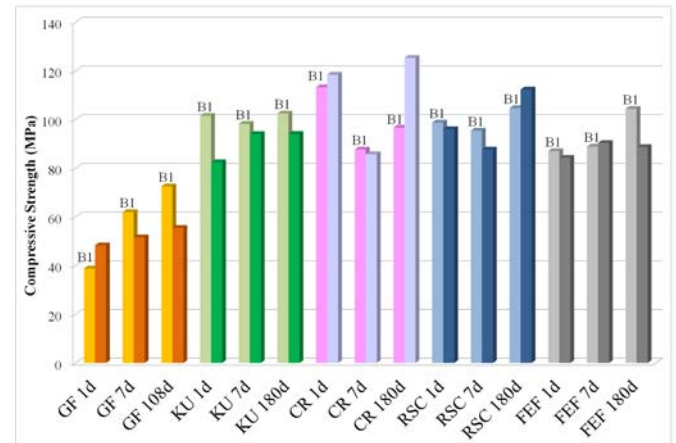**Figure 5.** Vickers hardness mean values of Batches over days 1, 7 and 180.

Figure 6. Compression strength mean values (MPa) for Batches over days 1, 7, 180.

from B1 and B2 were within the -10% to 10% range. However, the hypothesis that no significant difference will exist between the batches for the Vickers hardness and compressive strength were accepted for the days 1, 7 and 180. The PLR of GICs is pivotal to the strength and longevity of dental restorations (Fleming, 2003). A PLR variation from that suggested by the manufacturer for a Carbomer has not been assessed in any previous studies done on the GF Carbomer. This study however only focussed on the PLR differences between batches and their effect on the Vickers hardness and compressive strength.

The PLR has an effect not only on the physical properties of the material but also alters the setting time (Eames et al, 1977). A greater amount of powder and/ or less liquid decreases the setting time and increases the compressive strength. Decreases in PLR hinder various properties of the material and acid erosion of the restoration is more likely to occur (Zahra et al, 2011). The material has to resist the stresses that occur during mastication in posterior teeth (Dowling and Fleming, 2008), since this will be the area in which the material used in most clinical situations. When the optimal PLR relationship is maintained for the GICs and Carbomer materials, the material should perform as the manufacturer intended if the clinical scenario permits. The PLR will influence the way the GICs internally counters the compressive strength and elastic modulus during function (White and Yu et al., 1993). *In vitro* compressive strength provides insight into the properties of a material, since masticatory forces are partly compressive (Craig, 1997). Additionally, the surface microhardness parameter provides insight into the material's resistance to penetration of the surface. The recommended PLR, which appears on the manufacturers' packaging, is therefore the "gold standard" to which the capsulated materials' powder and liquid should adhere. Each manufacturer sets their own PLR in accordance with their internal research and development of the material. Subsequently, the manufacturers compile their internal testing data from this premise of the recommended PLR in the capsule.

The greater powder ratios in both GICs and RMGICs have resulted in accelerated setting reactions.

This therefore reduces the working time for the clinician (Quackenbush et al., 1998; Fleming et al., 2012). Inconsistencies in the PLR were observed between batches of the same GIC material and Carbomer in this study and PLR above the 10% of the manufacturer's recommendation result in an increased viscosity, which can influence the clinical handing. This was evident in a study where clinicians mixed GICs to their preference (Billington, 1990).

From the results of the present study, it was apparent that FEF had the most individual capsule observations with PLRs above 10% of the manufacturers' recommendations. It was expected that B1 and B2 from each material would have no difference in the PLR between the capsules from that manufacturer. This study illustrated that there were differences based on the SD between the batches of the materials in relation to the manufacturers' recommendation for the PLR of the GICs capsules. The spread of the observations are illustrated in Figure 4, but are only relevant if there are capsules with a PLR above the 10% perameter. How these variations of the PLR between the manufacturer's recommendation and the actual content of the PLR in the capsule influences the Vickers hardness and the compressive strength were determined to have no significant influence between the two batches for the same time periods. Variations between capsulated materials are expected, but the manufacturer placed an exact value in grams on the material brochures and not a range/estimated powder and liquid weight. There were various ranges presented in the literature on the permissible percentage that the PLR can vary below the manufacturer's recommendation before the properties are negatively affected (Fleming, 2003; Behr et al., 2008, Torabzadeh et al., 2011). The present study assessed the PLR in the capsules and illustrated that most observations were found to be above, but also within the 10% PLR recommended by the manufacturer. The literature on PLR mainly focused on the reduction of the PLR below that of the manufacturer's recommendation. The negative effects of too much liquid or too little powder resulted in a PLR value below that of the manufacturer's recommendation. The effective

reduction of the weight of powder that serves as the filler particle and the ion donor will reduce the ability to achieve an optimal compressive strength. GIC specimens from the literature were unable to withstand the in vitro analysis where the powder was kept constant and the liquid was increased to the level where the PLR was 17% below the manufacturer's recommendation (PLR -17%) (Behr et al., 2008). The physical properties of GIC specimens with various PLRs were assessed at PLRs of -10%, -20% and -50% powder to a constant volume of liquid as per the manufacturer's mixing recommendation. The -20% group presented with significantly lower compressive strengths and significantly longer setting times. The variation in the study of -10% PLR resulted in compressive strength variation not significantly different from the manufacturer's recommended PLR (Fleming, 2003) and was confirmed by this assessment of the GICs and Carbomer. All the aforementioned articles indicated that a PLR between -10% to 0% ratio of the manufacturer's recommendation allowed the compressive strength as well as the setting time to be within the material brochures of the manufacturer. It was concluded that in order to maintain the compressive strength of GICs, the PLR should not differ more than -10% to 10% from the manufacturer recommended PLR. An increase in the powder will result in a higher viscosity of the GICs, as noted during specimen production for this PLR study. The low Vickers hardness and compressive strength values of GF Carbomer could be because the specimen device could not accommodate the surface protective gloss and subsequent thermocuring with the LED application. The glass phase of the Carbomer has less network modifying ions available than regular aluminisilicate glass (Zainuddin et al., 2012). Another study concluded that the compressive strength was not significantly increased during the thermocure with the LED through the gloss (De Caluwe et al., 2017). Therefore, the gloss and LED thermocure that were not completed did not necessarily lead towards the lower compressive strength values.

Although there were differences in the individual weights of the powder and the liquid for the GICs and the Carbomer, the Vickers hardness and the compressive strength was not significantly affected.

The viscosity of some of the capsulated GICs and the Carbomer did not necessarily change, since the PLR could remain close to the manufacturer's recommended PLR, except for FEF where the extrusion difficulty was noticeable with most of the capsules. Based on Table 3, the net ratio between the powder and the liquid weight resulted in lower weights than those recommended by the manufacturer. Assessing Figure 4 and Table 4 the variation of -10% to 10% illustrated that most of the observations for CR, GF, IM, KU and RSC. Considering that the compressive strength had no significant difference, it would support this -10% to 10% PLR deviation from the manufacturer's PLR recommendation to be acceptable. The observations in Figure 4 of CR, KU and RSC B2 were the only materials well distributed around the manufacturer's recommendations. The SD between observations from a manufacturer represents

the significant difference in the spread between various observations of B1 and B2 in Table 4. Although the SD of the mean showed significant differences for some materials, all the SDs from a clinical and in vitro testing perspective were still well below the -10% to 10% variation of the mean PLR value. The p-values for the mean of B1 versus B2, as well as the SD of B1 versus B2 indicated that the filling of the capsules with powder and/ or liquid were relatively consistent and close together although the PLR was above that recommended by the manufacturer. The manufacturers consistently over-filled the capsules with powder and/or liquid with the exception of CR B1, CR B2, KU B1 and KU B2 (Figure 4). When Table 3 is considered for RSC B2 it becomes clear what a large role the powder and/or liquid weights play in the final PLR. Figure 4 illustrates the PLR of the 20 capsules of RSC B2 being well distributed around the manufacturer's PLR (only a +0.63% difference, Table 3). However, when the individual powder (-7.93%) and liquid (-8.57%) mean values for RSC B2 were below the weight stated by the manufacturer, the PLR was very close to the manufacturer PLR. KU B1 mean powder (-0.17%) and liquid (-0.66%) compared to RSC B2 further illustrated this individual powder and liquid importance (Table 3). Considering the consistently overfilled capsules, it was essential to evaluate the magnitude in terms of the effect on the PLR. Essentially the PLR is an important factor for the influence on the Vickers hardness and compressive strength. During the assessment of the PLR from B1 and B2, it became clear that if the mean PLR value of the capsules per batch was considered, CR, GF, IM, KU and RSC had mean PLR values that differed less than -10% to 10% from the manufacturer's PLR (Table 3). The majority of the FEF B1 and FEF B2 PLR were well above the manufacturer's PLR.

The further complexities of the PLR become clear when the various combinations of the powder and the liquid weight determine the PLR. The same mean PLR could have been achieved with both a high powder/ low liquid weight or vice versa. The B1 and B2 mean values as shown in Table 3 do not have large percentage differences when compared with the manufacturer's recommended PLR. Only FEF B1 (+10.79) and FEF B2 (+10.41) had a PLR higher than 10% of that recommended by the manufacturer. Table 3 shows the role the powder and the liquid play towards the resultant PLR in the capsules. This role explains the significant difference found between CR B1 and CR B2 as well as KU B1 and B2. The batches from CR and KU showed the best distribution of observations around the manufacturer's recommendation (Figure 4) as well as a relatively small percentage variation in relation to the other GIC powder and liquid weights. The mean value above/below the manufacturer's recommendation for CR B1 (-5.75) and CR B2 (-1.60) as well as for KU B1 (+0.52) and KU B2 (-0.94) were the reason for the significant difference between the batch mean PLR. There was a large difference between the GF powder and liquid content from the evaluated capsules compared to the manufacturer's recommendation (on the side of the packaging). Nevertheless, during statistical analysis of

the evaluated PLR, only a very small difference between the manufacturer's recommended PLR and the mean PLR of GF B1 (+6.32) and GF B2 (+5.02) were noted. This clearly illustrates the role in terms of statistical analysis where the combination of the powder and the liquid values can offset one another to have a PLR that is closer to the manufacturer's recommendation. Therefore, three items should be considered when assessing the effect of PLR in the capsules in relation to the manufacturer recommendation: 1) the internal data of the manufacturer is accepted as correct with the premise that the powder and the liquid are the same as the actual capsule PLR content, based on the provided individual powder and liquid weights. 2) the physical properties must not differ significantly. 3) a PLR range of -10% to 10% from the manufacturer's recommendation.

The literature further indicates that capsulated GICs have an advantage with their convenience of mixing, reduction in variation of the PLR and the ease of clinical application. Additionally capsulated GICs are mechanically mixed and therefore the mix is clinically reproducible.

Conclusion

Although the capsule PLR of the GIC materials can be well distributed around the manufacturer's PLR recommendation, the spread of the individual capsule PLR can influence the overall mean value. It is therefore essential that the -10% to 10% PLR be used to assess the individual GIC capsule for consistency

per manufacturer. The results concluded that no physical property with regard to Vickers hardness and compressive strength were negatively affected as no material had values below the -10% PLR. The small difference in the manufacturer's recommended mean values from the total observations in this study illustrated that GIC capsules are still a predictable dispensing format provided the manufacturer stays within a -10% to 10% range from their recommended PLR.

Limitations

IM could not be assessed for Vickers hardness nor compressive strength due to insufficient capsules per batch. The liquid content from the KU and IM groups were additionally confirmed by weight determination of the silver diaphragm as well. This could have also been completed for the plunger group as well for the liquid housing chamber.

Conflict of interests

The authors confirm that this article content has no conflict of interest.

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