

# COMPARISON OF THE DEGREE OF POWDER TO LIQUID RATIO VARIATION IN CAPSULATED GLASS IONOMER RESTORATIVE CEMENTS

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## ABSTRACT

### OBJECTIVE

The aim of this study was to assess the accuracy of the capsulated glass ionomer materials received from the dental material manufacturers. This was achieved by the comparison of the liquid to powder content as well as the variation of liquid to powder ratio of three capsulated glass ionomer restorative cements.

### MATERIALS AND METHODS

Three glass ionomer restorative cement materials were selected from the Paediatric dentistry clinic. The liquid content followed by the powder content was removed methodically and weighed on a chemical scale. 45 samples sealed material capsules were randomly removed from the bow of materials.

### RESULTS

KMA had the lowest average liquid (0.085g) and powder (0.288g) content with the results of the 15 KMA capsules closely grouped together. A statistical difference ( $P < 0.001$ ) was noted between the mean values of the liquids (CR, PGN, KMA), although the individual values had a wide spread. The powder variation was very small based on the spread. A statistical difference exist ( $P < 0.0001$ ) between the means of the recorded powder weights of CR (0.45g), PGN (0.37g) and KMA (0.29g).

### CONCLUSION

The decrease in the volume of the liquid could have various reactive possibilities with the powder. The handling and physical properties will be altered according to the liquid content at the time of titration of the GIC capsule. Manufacturers should ensure that the machines provide sufficient filling of the liquid chamber and ensure that no liquid is lost from the liquid chamber of the capsule, either by evaporation or capsular absorption before the materials' expire date.

### KEYWORDS

Glass ionomer, powder, liquid, capsule, physical properties.

Abbreviations and acronyms: GICs: Glass ionomer cements, Chemfil Rock: CR, ProGlass Nine: PGN, Ketac Molar Aplicap: KMA, standard deviation (SD)

## INTRODUCTION

The scientific problem that the clinician is faced with in daily dental practice is maintaining manufacturer instructions during the use of GIC dental materials. The liquid to powder ratio that clinicians maintain for glass ionomer cements (GICs) are pivotal to the strength and longevity of dental restorations. Glass ionomer restorative cements have relatively lower mechanical properties compared to composites, but form an important part in the dental material armamentarium. The favourable properties include the bond strength to moist tooth structure, thermal compatibility and the anti-cariogenic properties due to a release of fluoride.<sup>1</sup> GIC can be technique sensitive and some key disadvantages of these GICs may include low early strength and moisture sensitivity during setting.<sup>2</sup> The setting process of conventional GICs are characterised by an acid base reaction between the liquid and the powder. Therefore the liquid/powder ratios play an important role.<sup>3</sup> Initially GICs were hand mixed and articles related to the clinical manipulation by clinicians based on powder variation have been well documented.<sup>4,5</sup> Clinicians often mix GICs to a lower powder to liquid ratio than the manufacturers' recommendation. This has an affect not only on the physical properties of the material but also alter the setting time.<sup>3</sup> This leads to a weaker material with an altered setting time.<sup>6,7</sup> A decrease in powder liquid ratio may hinder the physical properties of the material and acid erosion of the restoration is more likely to occur.<sup>8</sup> This is a problem considering that the higher caries risk patient is especially indicated for a GIC. The material manufacturers launched the GICs in a pre-packed capsulated form in an attempt to maintain the physical properties and decrease operator variation in mixing the GICs.<sup>6</sup> The clinician can however still manipulate the titration speed and time of the capsulated GICs to increase or decrease the setting times. This could however result in a decreased liquid/powder ratio, especially at lower (3000osc) titration speeds.<sup>9</sup>

The aim of this study was to compare the liquid to powder content and the variation of liquid to powder ratio of three capsulated glass ionomer restorative cements as provided to the clinician by the dental material manufacturer.

## MATERIALS AND METHODS

Following ethical consideration for this *in vitro* study, the powder/liquid ratios in GIC capsules were performed. The liquid and the powder in the GIC capsules were assessed and compared to the corresponding samples from the same manufacturer. This was done to assess the accuracy and variability of the liquid and powder as received from the material manufacturer. Three GIC materials used regularly in the dental faculty were assessed. *Chemfil Rock* (DeTrey, Dentsply, Konstanz, Germany, Lot: 1310002003, 2016/08), *ProGlass Nine* (Silmet, Yehuda, Israel, Lot:1791022-A3, 2015/0400) , *Ketac Molar Aplicap* (3M ESPE, Minnesota, USA, Lot: 472606-A3, 2014/09) (Fig. 1).

#### Specimen preparation:

There were three test groups of GICs materials selected for powder/liquid determination for the GIC material capsules (Fig. 1). Fifteen samples were prepared for each of the three materials tested under standardised laboratory conditions. A single operator performed the weight determination of the liquid, followed by the powder weight determination at a constant room temperature ( $23\pm 1^\circ\text{C}$ ) with a relative humidity of  $50\pm 5\%$ .<sup>10</sup> The powder and the liquid were weighed on a desktop chemical scale (*Ohaus Precision Standard, Model TS400D, Ohaus Corp, Florham Park, N.J, USA*). Three identical pieces of filter paper 10mm wide and 10mm length (*70mm circle filter paper, Schleichter & Schüll, Germany, Lot:311608*) of a known weight was used to absorb all the liquid from the liquid holding chamber. Care was taken before and after every reading to “zero” the scale in order to ensure that the liquid and powder reading reflect the true value without the filter paper.

Chemfil Rock (CR, n = 15): The capsule was stored with the dispensing tip down for 30 minutes, allowing the liquid to pool in the bottom of the capsule. The grey plunger was removed carefully with a plier and wiped with the first filter paper (10 mm x 10 mm) to ensure that no liquid adhered to the plastic due to possible surface tension. The filter paper was held with a locking stainless steel tweezer and the liquid was absorbed from the holding chamber in the capsule. The second filter paper was used to absorb the liquid from the holding chamber. The third piece was used to ensure the holding chamber is free of any liquid. A visual inspection under 1.75 fluorescent magnifications (*Start International, Dallas, Texas*) was performed to ensure that no liquid remained in the chamber. The three pieces of paper was weighed and the liquid content calculated by deducting the papers’ starting weight with the final weight. The clear holding chamber was removed carefully and then a carbon surgical blade (*Swann-Morton, Sheffield, England*) was used to remove the thin plastic diaphragm, to expose the opening from where the powder would be expelled. The powder was placed on the 70 mm filter paper (of known weight). A visual inspection under magnification was performed to ensure that no powder was retained on the capsule walls due to static electricity. The 70 mm filter paper was weighed and the powder content calculated by deducting the papers’ starting weight with the final weight. The results were tabulated in a Microsoft excel<sup>®</sup> spreadsheet.

ProGlass Nine: (PGN, n = 15): The capsule was stored with the dispensing tip down for 30 minutes, allowing the liquid to pool in the bottom of the capsule. The purple plunger was removed carefully with a plier and wiped with the first filter paper (10 mm x 10 mm) to ensure that no liquid adhered to the plastic due to possible surface tension. The filter paper was held with a locking stainless steel tweezer and the liquid was absorbed from the holding chamber in the capsule. The second filter paper was used to absorb the liquid from the holding chamber. The third piece was used to ensure the holding chamber is free of any liquid. A visual inspection under

1.75 fluorescent magnifications (*Start International, Dallas, Texas*) was performed to ensure that no liquid remained in the chamber. The three pieces of paper was weighed and the liquid content calculated by deducting the papers' starting weight with the final weight. The black holding chamber was removed carefully and then a carbon surgical blade (*Swann-Morton, Sheffield, England*) was used to remove the thin plastic diaphragm, to expose the opening from where the powder would be expelled. The powder was placed on the 70 mm filter paper (of known weight). A visual inspection under magnification was performed to ensure that no powder was retained on the capsule walls due to static electricity. The 70 mm filter paper was weighed and the powder content calculated by deducting the papers' starting weight with the final weight. The results were tabulated in a Microsoft excel<sup>®</sup> spreadsheet.

Ketac Molar Aplicap: (KMA, n = 15): The capsule was not stored with the dispensing tip down for 30 minutes, since the liquid is packed in a silver diaphragm under the blue activation cover. This silver package was weighed with the liquid content. The KMA activator however removed all of the liquid from the silver package into an empty KMA capsule and discarded. It was noted that the silver foil weight was constant at 0.0412g. The liquid weight was obtained by deducting the start weight from the end weight after the KMA applicator was used. Subsequently the powder was removed from the specimen capsule after the brown plunger was removed carefully with a plier. The powder was placed on the 70 mm filter paper (of known weight). A visual inspection under 1.75 fluorescent magnifications (*Start International, Dallas, Texas*) was done to ensure that no powder was retained on the capsule walls. The 70 mm filter paper was weighed and the powder content calculated by deducting the filter papers' starting weight with the final weight. The results were tabulated in a Microsoft excel<sup>®</sup> spreadsheet.

After the capsules of the three materials were dismantled and the respective weights of the liquid and powder were determined all the products were disposed of accordingly.

## RESULTS AND DISCUSSION

The mean values and the standard deviation (SD) has been calculated for the liquid and powder of the three materials tested. The p-value summarize the results obtained from the one way ANOVA analysis of variance (not assuming equal variances) in the case of the mean values. The Bartlett test was performed on the variances (Table 1). There were significant differences ( $p < 0.0001$ ) between the means of the powder weights of the three materials (CR, PGN, KMA). Although there were no statistical difference between the mean values for the liquid, the

standard deviation values differed statistically between the three materials' liquid value ( $p < 0.0001$ ).

The differences between the mean values for the liquid (Fig. 2) and the powder (Fig. 4) were illustrated with a  $\pm 1.4$  (standard error) limit. The spread of the values recorded for the liquid of CR and PGN are similar, but KMA has a very small spread (Fig. 2). The scatter diagram also provides insight on the spread of the 15 individual liquid values and the average for each of the three materials (Fig. 3). The mean values of the liquid are not statistically different but the spread of the values are pronounced for CR and PGN compared to the spread of KMA (Fig. 2, 3). The powder variation was very small based on the spread. A statistical difference exist ( $P < 0.0001$ ) between the means of the recorded powder weights of CR (0.45g), PGN (0.37g) and KMA (0.29g) (Fig. 4, 5).

The recorded weights of the liquid and powder can be illustrated with all three materials on the same scatter plot (Fig. 6). These representations of the recorded weights provide insight into the spread of the paired liquid/powder weights. KMA is the material with the least spread of the paired liquid/powder. PGN and CR each have samples with either a very low or very high liquid content, but the powder content are in line with the greatest number of samples (Fig. 5, 6). The KMA has the lowest liquid and powder weight of the materials tested and according to the manufacture yields at least expressible 0.14g of material. A study by Dowling et al (2008), found no difference in the compressive strength and elastic modulus of KMA.<sup>11</sup> This result correlates well with the fact that all the samples of KMA was above the 80% mark (Fig. 7)<sup>7</sup>.

The liquid of the Chemfil Rock consist of polycarboxylic acid (10-25%) and tartaric acid (2.5-10%) and water with an unknown percentage resulting in a pH of 2.5. The powder phase contains a zinc modified fluoro-alumino-silicate glass filler particles and polycarboxylic acid crystals (10-25%).<sup>12</sup>

ProGlass Nine has a liquid phase contains polyacrylic acid (30-40%), tartaric acid (5-10%) and distilled water (50%). The powder contains Alumino-silicate glass (90-100%) and polyacrylic acid crystals (5-10%).<sup>13</sup>

Ketac Molar has a liquid phase of water (60-65%), copolymer of acrylic acid-maleic acid (30-40%) and tartaric acid (5-10%). The powder has a glass powder of unknown content (possibly 93-98%, trade secret), copolymer of acrylic acid-maleic acid crystals (possibly 1-5%, trade secret).<sup>14</sup>

It is imperative that the correct liquid powder relationship is maintained for GIC materials since the material has to resist the stresses that occur in posterior teeth.<sup>11</sup> These stresses are countered by the materials compressive strength and the elastic modulus.<sup>15</sup> The decrease in the volume of the liquid phase could have various reactive possibilities with the powder and therefore alter the compressive strength and the elastic modulus. In the first instance the acid component could be normal and the water content decreased. Secondly the water content could be normal and the acid content decreased. Thirdly a combination of acid and/or water concentrations could vary.

When the clinician mixes the GI capsules in the amalgamator at the appropriate speed the first stage of the setting reaction starts. The first stage of the liquid powder mixing is the water and polycarboxylic acid that hydrate the glass

particle as well as the polycarboxylic acid crystals in the powder. Under ideal circumstances there is an exchange of protons from the glass filler particle causing the release of the cations ( $Zn^{2+}$ ,  $Ca^{2+}$ ,  $Al^{3+}$ ,  $Sr^{2+}$ ). While these cations are released from the glass particle the water in the liquid cause the polycarboxylic acid in the liquid and powder phase to neutralize and form a  $COOH^-$  molecule. The initial cations ( $Zn^{2+}$ ,  $Ca^{2+}$ ,  $Al^{3+}$ ,  $Sr^{2+}$ ) that were released from the glass particle will cross link ionically to the  $COOH^-$  resulting in the “salt bridge” formation.<sup>16</sup>

In the first possible scenario for the materials PGN and CR: if the water content in the liquid is less than what is required to neutralize the polycarboxylic acid, the “salt bridge” formation will be very rapid due to the acids causing an increased release of cations from the glass particle. The decreased water content will additionally not be able to neutralize the  $COOH^-$  that forms due to the polycarboxylic acid liquid as well as the acid from the powder phase. The material will therefore set much faster than the prescribed manufacturer’s time.

Should the water content be adequate as in the second possible scenario the viscosity of the tartaric- and polycarboxylic (CR) or tartaric- and polyacrylic acid (PGN) will be “diluted” in the relatively higher water content and a decreased cation release from the glass particle will occur. The  $COOH^-$  molecules will be neutralizes pre-maturely and the setting reaction will be increased and a decreased surface hardness will be present.

The third possibility of where a combination of acid and/or water concentrations could vary the anticipated reaction on the glass particles and  $COOH^-$  molecule becomes erratic and difficult to establish the exact outcome of the materials handling.

Fleming et al (2003), performed a study with a hand mixed GIC, assessing the physical properties with various powder ratios to a constant liquid ratio that was maintained at 1g. This study found that powder ratios 50% and 80% less than the manufacturer instructions statistically decreased the compressive strength as well as a decrease in the setting time of the GIC tested.<sup>7</sup>This decrease in powder leads to a decreased concentration of reinforcing glass particles, which results in a decreased load bearing capacity.

The manufacturers do not state the liquid/powder ratio and weight of either in the capsules. The instructions of PGN powder/liquid hand mix kits comply with ISO 9917:2003 and advise a powder to liquid ratio of 4.1:1.0. This is obtained by measuring one level scoop and mixing it with one drop of liquid. In order to prevent an incorrect deduction as stated in the idiom: “Comparing apples with oranges” the results obtained from the three materials must be converted to the same denomination of liquid and the appropriate ratio maintained to convert the powder. By doing the conversion the capsule results could be comparable to the studies done in the literature.<sup>5,7,17</sup> The liquid weight obtained from all 45 samples and the respective averages was converted to 1g values and the respective liquid/powder ratios maintained (Fig. 7).<sup>7</sup> It is imperative that the clinician note that because the manufacturers do not provide the adequate liquid/powder ratio information regarding the content of the capsule, the “average” of the 15 samples for each group was selected as the “adequate” liquid/powder ratio and applied to

the 50% and 80% results obtained by Fleming et al (2003). The result from this conversion of the liquid to 1g and the appropriate ratio applied to the powder was that no powder values were present at the 50% reduction in powder (1.99g) but there were several samples for CR and PGN that had an 80% reduction in powder to the 1g liquid. KMA had no values below the 80% powder ratio based on the average. PGN and KMA on the other hand will have more capsules than CR setting faster than the “average” for that material respectively (Fig. 7). If the powder is more than what the liquid can hydrate a faster setting reaction will take place and the material will set, before the restoration is placed in the prepared cavity.<sup>7</sup> If the hand mix ratio of PGN is honoured and provided the PGN capsule powder has no modification compared to the hand mix powder of PGN then it is noted that the powder liquid ratio in the capsule should have been 5.57g powder to 1g of liquid rather than the obtained “average” of 3.98g powder to 1g liquid. This value of 5.57:1 will translate into only one sample being above the 80% threshold and the other 14 samples will then have inferior compressive strength (Fig. 7).

In conclusion, the liquid to powder ratio and variability will affect the clinical handling and physical properties of the GIC.<sup>8</sup> The literature has shown that capsular GIC stay superior to hand mixing due to a decrease in operator variability and ease of application into the cavity preparation.<sup>18</sup> The liquid and powder ratio must be accurately maintained during the manufacturing process. The liquid phase however, has to be packaged by the manufacturer in a way that prevents the liquid from evaporation and/or penetration into the plastic of the capsule over time. This limitation of the liquid variation is important since it has been established that capsule GIC are more reliable in terms of providing an increased compressive fracture strength and elastic modulus compared with hand mix materials.<sup>11</sup>

Although there was a large spread of the liquid data obtained from the capsules, it was established that only five of the 45 samples had a powder/liquid ratio below the 80% mark<sup>7</sup> (in accordance with an “average”) and would therefore have a potentially lower compressive strength and an increased setting time. The individual packaging of the KMA liquid in the silver foil may add an additional piece of activator equipment, but it will remove the variability of the liquid contents more effectively than the liquid chamber of CR and PGN. This in turn will reduce the variation in the setting reaction, physical properties and void distribution within the final restoration.

This study illustrated that there is a large variation in the liquid and powder ratios between capsules from the same manufacturer. Therefore, non-invasive future research must be completed to assess the ratio of liquid/powder with Micro 3D ct-reconstruction. The liquid:powder ratio determination can then be followed by the determination of expressible yield and physical properties of the capsulated GIC. This will provide a clear insight to how the variation of the liquid:powder ratios from the manufacturer influence the results of the GIC physical properties.

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## FIGURE LEGENDS

Figure. 1. GIC materials and Filter paper

Figure. 2. Mean value of the liquid for the three GICs

Figure. 3. Scatter diagram of the 15 individual liquid weights.

Figure. 4. Mean value of the powder for the three GICs

Figure. 5. Scatter diagram of the 15 individual powder weights.

Figure. 6. Liquid to powder ratio for each of the 15 Samples from the three materials.

Figure. 7. Liquid/powder ratio converted for liquid at 1g.

## FIGURES AND TABLES

Material	Powder		Liquid	
	Mean	SD	Mean	SD
CR	0.4548	0.017	0.1145	0.0291
PRN	0.3695	0.0143	0.0928	0.0254
KMA	0.2885	0.0078	0.0852	0.0011
p-value	<0.0001	0.0227	0.0002	<0.0001

Table 1: Mean and standard deviation values of the liquid and powder of three GIC materials.



Figure. 1. GIS materials

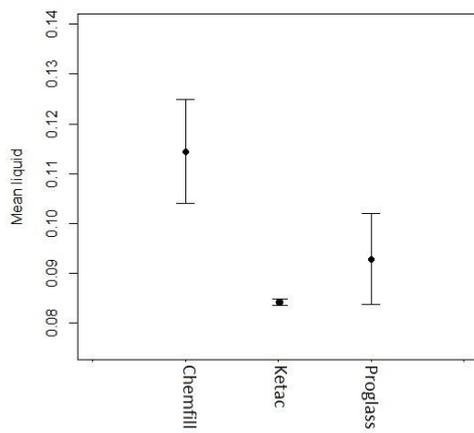


Figure. 2. Mean value of the liquid for the three GIC materials.

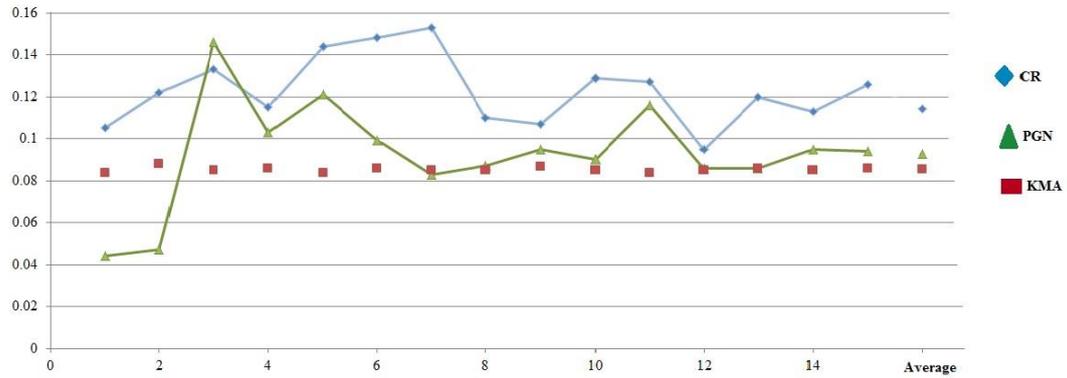


Figure 3. Scatter diagram of the 15 individual liquid weights.

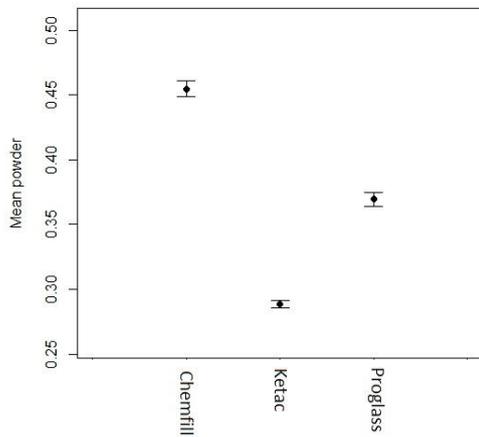


Figure 4. Mean value of the powder for the three GICs

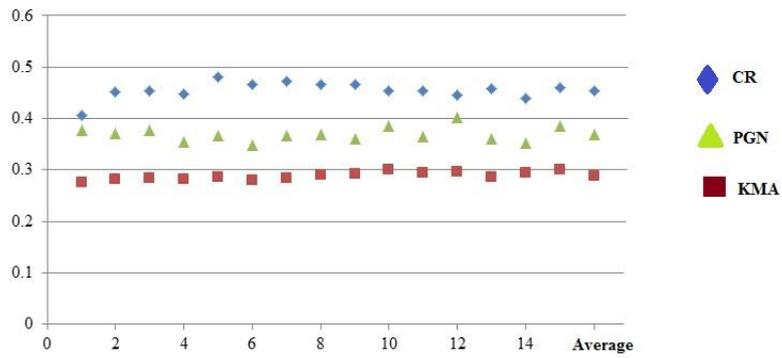


Figure 5. Scatter diagram of the 15 individual liquid weights.

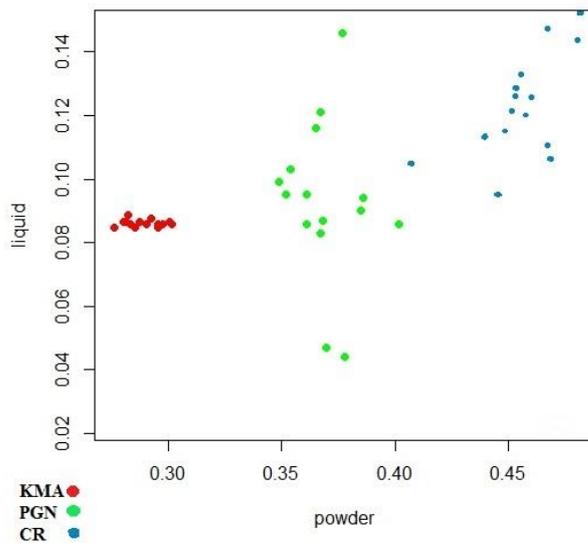


Figure 6. Liquid to powder ratio for each of the 15 Samples from the three materials.

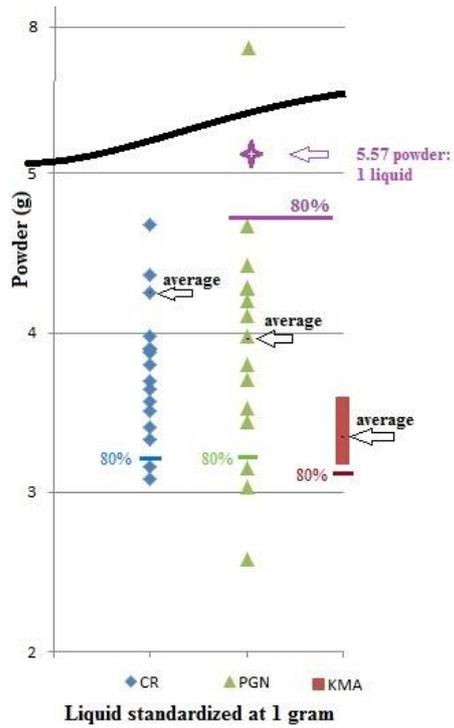


Figure 7. Liquid/powder ratio converted for liquid at 1g.