POWDER-LIQUID RATIO AND PROPERTIES OF TWO RESTORATIVE GLASS IONOMER CEMENTS

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ABSTRACT

Changes in the powder-liquid ratio of glass ionomer cements may affect some of its physical properties and acid erosion.

The aim of this study was to evaluate the physical properties and acid erosion of two conventional restorative glass ionomer cements against ISO 9917-1:2007 standards after changing the powder-liquid ratio to an adequate consistency for luting indirect restorations.

The methodology of ISO Specification 9917-1:2007 was applied to the powder-liquid ratio indicated by the manufacturer and to a modified ratio. Two restorative glass ionomer cements, ChemFil (Ch) (Dentsply) and Ionofil Plus (IP) (Voco), were used to evaluate film thickness, compressive strength, net setting time and acid erosion. Thickness was measured three times with a digital micrometer (Digimatic Mitutoyo Corporation). Sample size was five for each cement or condition. Compressive strength (Instron 1011, crosshead speed of 1 mm/min) was evaluated after 24 h immersion in water at 37°C. Sample size was five for each cement or condition. Setting time was evaluated for Ch and IP at 37°C. Sample size was three for each cement or condition. Specimen moulds (30 x 30 x 5 mm) with a central perforation of 5 mm in diameter and 2 mm depth were used for acid erosion tests. Erosion depth was measured with a micrometer gauge with a precision of 0.001 mm, before and after 24-hour immersion in a lactic acid-sodium lactate solution with pH 2.74 at 37°C. Sample size was five for each condition. Student's t test was performed with a level of significance of $p < 0.05$ for each material and condition tested. Arithmetic mean (Standard Deviation). Powder-liquid ratio according to manufacturers: film thickness (in µm): Ch 220 (40), IP: 382 (5); compressive strength (in MPa) at 24 hs: Ch 166.3 (16,6), IP: 100 (10); net setting time (in min.) at 37°C: Ch 3.44 (0.3), IP: 5.26 (0.1) ; depth of acid erosion (in mm): Ch 0.15 (0.02), IP: 0.17 (0.02). Modified powder-liquid ratio: film thickness (in µm): Ch 23(1), IP:24(1); compressive strength at 24 hs (in MPa): Ch: 69.3 (14.6), IP: 46.5 (7.4); net setting time (in min.) at 37°C: Ch 5.72 (0.1) and IP 9.38 (0.1); depth of acid erosion (in mm): Ch 0.22 (0.02). Data were not recorded for IP because the sample disintegrated in the solution.

Student's t test was performed for both materials and conditions with a level of significance of $p < 0.05$. The difference between each condition tested was statistically significant ($p < 0.01$).

While changes in the powder-liquid ratio of a restorative glass ionomer cement can result in some of its properties having values that are not far from those required for luting cements according to ISO specifications, it did not meet the requirements for acid erosion.

Key words: polyalkenoate cement, physical properties, solubility.

RELACIÓN POLVO-LÍQUIDO Y PROPIEDADES DE DOS IONÓMEROS VÍTREOS DE RESTAURACIÓN

RESUMEN

Las modificaciones en la relación polvo-líquido de los cementos de ionómero vítreo podrían afectar algunas de sus propiedades físicas y su erosión ácida.

El objetivo de este trabajo fue evaluar las propiedades físicas y la erosión ácida de dos cementos de ionómero vítreo para restauración, según la Norma ISO 9917-1:2007, luego de modificar la relación polvo-líquido alcanzando una consistencia adecuada para fijar restauraciones rígidas.

Se aplicó la metodología de la Norma ISO con una relación polvo-líquido según indica el fabricante y una relación modificada. Se evaluó espesor de película, resistencia compresiva, tiempo de fraguado y erosión ácida en dos cementos de ionómero vítreo para restauración: ChemFil (Ch) (Dentsply), Ionofil Plus (IP) (Voco). El espesor de película fue determinado con un micrómetro digital (Digimatic Mitutoyo Corporation). El tamaño de la muestra fue de 5 unidades para cada cemento o condición experimental. La resistencia compresiva (Instron 1011, velocidad del cabezal 1 mm/min) fue evaluada luego de la inmersión en agua destilada a 37°C. n=5. El tiempo de fraguado se evaluó a 37°C. n=3. Se utilizaron soportes de probeta (30 x 30 x 5 mm) con una perforación central de 5 mm de diámetro y 2 mm de profundidad para los ensayos de erosión ácida. La lectura de la profundidad de erosión de las probetas fue realizada con un calibre con 0.001 mm de precisión, antes y después de la inmersión durante 24 horas en una solución de ácido láctico-lactato de sodio con pH 2.74 en estufa a 37°C. n=5. La prueba de Student fue realizada con un nivel de significancia de $p < 0.05$ para cada material y condición.

Media Aritmética (Desviacion Estándar) Relación polvo-líquido según el fabricante: espesor de película (en µm): Ch 220 (40), IP: 382 (5); resistencia compresiva (en MPa) a las 24 hs: Ch 166.3 (16,6), IP: 100 (10); tiempo de fraguado (en min.) a 37°C: Ch 3.44 (0.3), IP: 5.26 (0.1) ; profundidad de erosión (en mm): Ch 0.15 (0.02), IP: 0.17 (0.02). Relación polvo-liquido modificada: espesor de película (en µm): Ch 23(1), IP 24(1); Resistencia *compresiva a las 24 hs (en MPa): Ch: 69.3 (14.6), IP: 46.5 (7.4); tiempo de fraguado (en min.) a 37°C: Ch 5.72 (0.1), IP 9.38 (0.1); profundidad de erosión (en mm): Ch 0.22 (0.02). No se registraron resultados para IP pues las muestras fueron desintegradas por la solución. La Prueba de Student fue realizada para los dos materiales y condiciones experimentales con un nivel de significancia de p< 0.05. La diferencia entre cada condición de ensayo fue estadísticamente significativa (p<0.01).*

Las modificaciones de la relación polvo-líquido introducidas, pueden acercar las propiedades finales de un ionómero vítreo para restauración a lo requerido para uno de fijación según las especificaciones de la Norma ISO, a excepción de los requerimientos para erosión ácida.

Palabras clave: cemento polialquenoico, propiedades físicas, solubilidad.

INTRODUCTION

A wide variety of dental cements are used in clinical practice to restore, lute or seal[1]. Conventional cements – like glass ionomers – rely on an acid-base reaction with formation of an ionic salt and generate a molecular adhesion to tooth structures. These materials can be supplied either in capsules or in a hand mix version (powder and liquid) that usually comes with a dispenser for preparing the correct powder-liquid ratio for the specific use designed by the manufacturers[2]. Many studies have shown that the amount of powder used changes between one operator and another, and it is presumed that changes in the powder-liquid ratio of glass ionomer cements can affect some of its physical properties [3] and acid erosion. Nevertheless, many practitioners deliberately change the powder-liquid ratio to obtain an adequate consistency for luting a restoration with glass ionomer cement. Clinical success is related to luting procedures as well as to the intrinsic characteristics of the luting material[4].

Any variation in the powder-liquid ratio will affect the mechanical properties of some cements[1]. In some commercial products, the hand mix glass ionomer cements are difficult to handle because neither the scoop nor the liquid dropper contain constant weight or volume. Although an accurate mixture of cement is essential, disparities occur[3]. Conventional glass ionomer cements are more sensitive in their reaction to higher liquid content than to higher powder content, and mixing variations happen even in cases where portioning aids were used[3].

The manufacturer's specifications, including the powder-liquid ratio, are intended to produce minimal modifications in the final properties of the cement[5].

Film thickness is influenced by manipulative variables such as mixing temperature and powder-liquid ratio[1,6]. One of the main features that affects it directly is the consistency of the luting material[5,7-9]. Depending on the particular clinical situation, the consistency of the material may adversely affect the film thickness and the correct seating of the restoration[7,9]. A luting material with increased viscosity requires more time to reach the optimal seating of the restoration before it sets as well as the application of higher seating forces to prevent marginal gaps[7].

Compressive strength is the most commonly used strength value to characterize dental cements[10]. In glass ionomer cements, it continues to increase over several weeks to about 200 MPa. This is thought to be due to reconstruction of a silicate network[1].

Any restorative dental materials must withstand the functional forces and exposure to various media in the mouth to achieve optimal clinical performance over a considerable period of time[10].

The aim of this study was to evaluate the film thickness, compressive strength, net setting time and acid erosion of two conventional restorative glass ionomer cements against the ISO 9917: 2003 requeriments, after changing the powder-liquid ratio to adequate consistency for luting indirect restorations.

MATERIAL AND METHODS

Tests were conducted under standarized temperature (21ºC ± 2ºC) and humidity (60% ± 10%). Table 1 shows the materials selected for the experiments with a powder-liquid ratio as indicated by the manufacturers and a modified ratio. Two restorative glass ionomer cements were used to evaluate film thickness, compressive strength, net setting time and acid erosion: ChemFil (Ch) (Dentsply) and Ionofil Plus (IP) (Voco GmbH). The methodology established by the ISO Specifications 9917-1:2007 for glass ionomer cements was used, except for film thickness, where the specification was adapted[11] (Table 2).

Thickness was measured three times to the nearest 0.1 μm with a digital micrometer (Digimatic Mitutoyo Corporation) (Reading A).

A measured amount equivalent to 0.1 ml of each cement mixture was placed between two glass plates (12 x 12 x 5). A 25 N load was applied on the upper glass plate with a hydraulic machine (CIFIC, Rosario,

Table 1. Products and Powder-Liquid Ratio used.

Type of Cement	Trade Name	Code	Powder - Liquid Ratio	Manufacturer	Batch
Restorative glass ionomer	ChemFil	Ch	Manufacturer: 7.4:1 Modified: 3.7:1	Dentsply	489085
Restorative glass ionomer	Ionofil Plus	IP	Manufacturer: 5.15:1 Modified: 2.57:1	Voco	P: 611188 L: 601462

Table 2. ISO 9917-1:2007 Requirements for Glass Ionomer Cements.

Type of material	Film Thickness (μm)	Compressive strength (MPa)	Net setting time (minutes)		Acid erosion (mm)
	max.	min.	min.	max.	max.
Restorative	-	100	1.5	6	0.17
Luting	25	50	1.5	8	0.17

Argentina). Ten minutes later, the overall thickness of the plates and the cement was recorded (Reading B). The difference between readings A and B (difference between the thickness of the plates with and without the material between them) was considered as the final combined film thickness for the specimen being tested. Sample size was five for each cement or condition. For each cement, 4 x 5 mm cylinders were made in stainless steel moulds. Compressive strength was evaluated after 24h immersion in distilled water at 37ºC, with an Instron Machine 1011, with a crosshead speed of 1 mm/min. Values for compressive strength were obtained in MPa by relating the application force (N) to the cylinder surface (mm^2). Sample size was five for each cement or condition.

Setting time was evaluated for Chemfil and Ionofil Plus at 37ºC. One tenth of a milliliter of the cement was placed in a metal mould with a central perforation of 10 x 6 mm. Ninety seconds after the end of mixing, a metallic indentor with a flat end 1 mm in diameter was applied vertically onto the surface of the cement for 5 seconds**.** The indentations were repeated at thirty-second intervals (in the first case) and at ten-second intervals in the others, until the needle failed to make a complete circular indentation in the cement. The time elapsed between the end of mixing and the time when the needle failed to indent the material was considered as setting time for the material tested. Sample size was three for each cement or condition.

Acid erosion was determined using 30 x 30 x 5 mm specimen moulds with a central perforation of 5 mm in diameter and 2 mm depth. Erosion depth was measured with a micrometer gauge with a precision of 0.001 mm, before and after 24 hour immersion in a lactic acid-sodium lactate solution with pH 2.74 at 37ºC. Both materials were tested with a powder-liquid ratio as indicated by the manufacturer and a modified ratio. Sample size was five for each condition and experimental conditions were compared using Student's t test.

RESULTS

Table 3 shows means and standard deviations as well their 95% confidence intervals for the film thickness, compressive strength, setting time and acid erosion of the glass ionomer cements tested, with a powder-liquid ratio as indicated by the manufacturers and a modified ratio.

Student's t test was performed for each material and condition with a level of significance of $p< 0.05$. The difference between each condition tested was statistically significant ($p<0.01$) for film thickness and compressive strength as shown by the lack of overlap of the confidence intervals. When the powder-liquid ratio of Ionofil Plus was modified, it did not meet the minimum required compressive strength specification (50 MPa).

Setting time was studied at 37°C with a powder-liquid ratio according to manufacturers and a modified ratio. Ionofil Plus did not meet the ISO Specification requirements for a luting cement when the powder-liquid ratio was modified, because it exceeded the 8 minutes maximum expected (9.38 min). Student's t test showed that the difference between each material and condition tested was statistically significant ($p<0.01$), and that there were statistical differences for both materials because of the lack of overlap in the confidence intervals.

Table 3. Properties of Glass Ionomer Used.

Material	Properties	P/L Ratio	AM	SD	CI (95%)
ChemFil	Film Thickness (μm)	Manufacturer	220.0	40.0	171 – 270
		Modified	23.0	1.0	22 – 24
	Compressive Strength (MPa)	Manufacturer	166.3	16.6	146 – 187
		Modified	69.3	14.6	51 – 87
	Setting Time (min)	Manufacturer	3.44	0.3	2.8 – 4.1
		Modified	5.72	0.1	5.5 – 6
	Acid Erosion	Manufacturer	0.15	0.02	0.13 – 0.17
		Modified	0.22	0.02	0.20 – 0.24
Ionofil Plus	Film Thickness (μm)	Manufacturer	382.0	5.0	376 – 388
		Modified	24.0	1.0	23 – 25
	Compressive Strength (MPa)	Manufacturer	100.0	10.0	88 – 112
		Modified	46.5	7.4	37 – 56
	Setting Time (min)	Manufacturer	5.26	0.1	5.0 – 5.5
		Modified	9.38	0.1	9.1 – 9.6
	Acid Erosion	Manufacturer	0.17	0.02	0.15 – 0.19
		Modified	NO DATA		

The depth of acid erosion (mean and standard deviation in mm) was evaluated for both materials. Student's t test showed that the difference between each condition tested for Chemfil was statistically significant ($p<0.01$). Chemfil did not meet the requirements of ISO 9917-1:2007 when the powder-liquid ratio was modified. Data were not recorded for Ionofil Plus specimens when the powder-liquid ratio was modified because the samples disintegrated in the solution.

DISCUSSION

Conventional glass ionomer cements are frequently used in dental practice to restore or lute restorations of dental and other structures[1,4] because of its advantages, such as fluoride ion release, physicochemical bonding to tooth structure and a low coefficient of thermal expansion[7].

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[7].

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[1,2,6,8,9], including manipulation, temperature of the mix, consistency, handling, working and setting time and time elapsed after mixing[1,3,-6,8]. Consistency is considered one of the principal features affecting the film thickness of any luting material.[8] In this study, when the powder-liquid ratio was modified to adequate consistency for a luting cement, both materials tested, Chemfil and Ionofil Plus met the 25 µm maximum required by ISO 9917, as shown by the lack of overlap of the confidence intervals.

Compressive strength has been used as a predictor of clinical performance. For a restoration to function correctly, the cement must have sufficient strength to resist fracture[1]. Both materials tested met ISO specifications regarding compressive strength when the powder-liquid ratio was used according to manufacturers. On the other hand, Ionofil Plus did not fulfill the requeriments when the modified powder-liquid ratio was used because it did not reach the minimum value of 50 MPa. One study has shown that the compressive strength of encapsulated glass ionomer cements which have high viscosity produce more favourable mechanical properties.[3] The reason was the lower probability of air inclusions during the mechanical mixing[4]. In contrast, more fluid materials produced lower porosity by hand mixing than by mechanical mixing. It appears that the lower viscosity material more readily causes air inclusions and a type of froth is formed during the rapid mixing process. The slower hand-mixing process helps to avoid these inclusions and may cause the collapse of the air-bubbles[3].

Net setting time was evaluated at 37ºC. Both Chemfil and Ionofil Plus took longer to set (double) when the powder-liquid ratio was modified. This was more noticeable in Ionofil Plus. The setting time of luting agents is influenced by temperature. As temperature increases, the working and setting times of glass ionomer cements decrease[1]. Although at 37ºC (according ISO 9917) the net setting time of any luting material is expected to be shorter than 8 minutes, Ionofil Plus exceeded the specification values.

Different strategies have been developed to obtain success in cementation because it depends on multiple factors[1,5,8,9]. The luting procedure has the potential to adversely influence marginal adaptation[9], so the ability of the cement to withstand exposure to different media in the mouth[10], to avoid water sorption and dissolution, is essential to clinical success[4]. Acid erosion tests were carried out for both materials and conditions, but the results did not meet the 0.17 mm maximum required for luting materials according ISO Specifications. Ionofil Plus specimens with the modified powder-liquid ratio disintegrated a few minutes after immersion in the lactic acid-sodium lactate solution.

Moreover, the acidity of the storage medium is not the only factor responsible for the degradation of cements. The ability to resist dissolution has been found to vary with the composition of the medium, and not simply with its pH[10]. The acid erosion tests provided an idea of the performance of the two restorative glass ionomer cements selected at adequate consistencies for luting restorations.

There is not a single specific material that could be used for the entire range of procedures, but the correct luting agent must be selected according to its mechanical and physical properties in order to ensure high clinical performance[1,2,4,7,8].

CONCLUSION

It can be concluded that, while changes introduced in the powder-liquid ratio of a restorative glass ionomer cement can result in values for some properties that are not far from those required for luting cements, the requirements of ISO specifications for acid erosion could not be met.

ACKNOWLEDGEMENTS

Financed within Project O009, Project O011 and with a scholarship granted to the first author by the University of Buenos Aires, Argentina.

The authors would like to thank Dentsply Argentina and especially Mr. Raúl Ripoll (Country Manager) for the materials and their profiles used in this paper, Dr. Delia Takara for her support in carrying out acid erosion tests, Dr. Analía Mosquera for her assistance with some tests and Mr. Javier Faig for his support with the tests done in this study.

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