Eur. J. Prosthodont. Rest. Dent., Vol.18, No. 4, pp 150-154
Printed in Great Britain

doi:10.1922/EJPRD_847Fleming05

Extrusion-Mixing Compared with Hand-Mixing of Polyether Impression Materials ?

Caroline McMahon*, Daniel Kinsella† and Garry J.P. Fleming‡

Abstract - *The hypotheses tested were two-fold (a) whether altering the base:catalyst ratio influences working time, elastic recovery and strain in compression properties of a hand-mixed polyether impression material and (b) whether an extrusion-mixed polyether impression material would have a significant advantage over a hand-mixed polyether impression material mixed to the optimum base:catalyst ratio. The polyether was hand-mixed at the optimum (manufacturers recommended) base:catalyst ratios (7:1) and further groups were made by increasing or decreasing the catalyst length by 25%. Additionally specimens were also made from an extrusion-mixed polyether impression material and compared with the optimum hand-mixed base:catalyst ratio. A penetrometer assembly was used to measure the working time (n=5). Five cylindrical specimens for each hand-mixed and extrusion mixed group investigated were employed for elastic recovery and strain in compression testing. Hand-mixing polyether impression materials with 25% more catalyst than that recommended significantly decreased the working time while hand-mixing with 25% less catalyst than that recommended significantly increased the strain in compression. The extrusion-mixed polyether impression material provided similar working time, elastic recovery and strain in compression to the hand-mixed polyether mixed at the optimum base:catalyst ratio.*

KEY WORDS: base:catalyst ratio, polyether impression materials

INTRODUCTION

Impression materials are used in dentistry to provide a detailed replicate of the dental anatomy and tissues to enable an accurate dental cast for the construction of well-fitting indirect restorations to be produced[1]. The rheological requirements for a satisfactory impression material include suitable flow and plasticity to record fine detail with sufficient working time to allow the clinician to mix, load and transport the tray to the patient's mouth. Ideally no change in viscosity during working should occur, however, a rapid increase during setting, to decrease the time the impression material spends in the mouth, is essential[2]. The set material should experience negligible dimensional changes during or after removal from the mouth and have sufficient elasticity to overcome undercuts without under-going permanent deformation[2]. Impression materials can be categorised as rigid, elastic and elastomeric. Rigid impression materials include impression compound which is a thermoplastic material that becomes soft and 'takes on a new form' above the glass transition temperature (T_g) of 55-60°C but cannot be removed from deep undercuts[2]. Elastic impression materials are viscoelastic and include hydrocollides which are characterised by the importance that the tray is removed in a 'rapid snap-action' so that a near elastic response occurs[2]. Elastomeric impression materials are characterised by polymers which are used above T_g and become more fluid at increasing temperatures above T_g [2]. Elastomeric impression materials are considered superior for dentate patients as they posses the ability for large scale recoverable deformation and long term dimensional accuracy[1,3].

Polyether impression materials were the first material to be developed specifically for the dental profession in the late 1970's[3], and are the most hydrophyllic of the elastomeric impression materials[4,5]. Polyether impression materials are routinely supplied in two forms, hand-mixed and extrusion-mixed. The manufacturers of hand-mixed polyether impression materials recommend that equal lengths of base and catalyst be used by operators to obtain the optimum base:catalyst ratio. However, hand-mixing has the potential to introduce variability to the base:catalyst ratio employed when dispensing the equal lengths of base and catalyst. Additionally, the mixing technique and mixing time are also susceptible to variability during mixing which can impact upon the functional characteristics of the hand-mixed polyether although mixing technique and mixing time were beyond the remit of the current investigation. With the introduction of extrusion-mixed polyether impression materials possible variability on dispensing the lengths of base and catalyst is eliminated by the use of pre-measured proportions of the constituents delivered through a special mixing tip.

The International Standard for Elastomeric Impression Materials[6] includes a range of testing methodologies employed to assess elastomeric impression material performance including working time, elastic recovery and strain in compression. The hypotheses tested in the current study were two-fold: (a) whether altering the base:catalyst ratio influences the working time, elastic recovery and strain in compression properties of a hand-mixed polyether impression material and (b) whether an extrusion-mixed polyether impression material would have a significant advantage

* B Dent. Tech
† Adv. City & Guilds (Crown and Bridge)
‡ BSc, PhD, ILTHE, FADM, FTCD

over a hand-mixed polyether impression material mixed to the correct base:catalyst ratio.

MATERIALS AND METHODS

The hand-mixed (Impregum™ F; 3M ESPE, Seefeld, Germany) and extrusion-mixed (Impregum™ Penta™; 3M ESPE, Seefeld, Germany) polyether impression materials investigated were purchased from the manufacturer. Interestingly in line with hand-mixed and mechanically mixed dental cement and dental restorative systems routinely used in dentistry, the manufacturers modified the base:catalyst ratio of the hand-mixed (7:1) and extrusion-mixed (5:1) materials to compensate for the differing mixing conditions. The differences between the hand-mixed and extrusion-mixed polyether impression materials investigated highlight that they cannot be considered identical in terms of chemistry in the current study.

Working Time

To asses the impact of working time, five specimens were prepared in accordance with the manufacturers recommendations (at a base:catalyst ratio of 7:1). The hand-mixed polyether was laid out in equal 9 cm lines of base and catalyst onto a mixing block. The two pastes were mixed together for 45 s (recommended mixing time) with a clean spatula and the resultant mix was placed into a cylindrical mould (25 mm height and 25 mm diameter) [7]. An acetate strip and glass plate were pressed on top of the mould to extrude any excess material, thereby creating a flat surface on top of the specimen. The surface of the specimen was dabbed with a penetrometer assembly at a force of 500 N using a stainless steel needle at 10 s intervals until the material pulled 1 cm away from the surface, indicating that the material was no longer pliable and the working time was obtained[7]. The procedure was repeated a further four times to provide a consistent measurement (n=5) for the working time of the hand-mixed polyether impression material investigated.

Additionally, further specimen groups with catalyst lengths increased and decreased by 25% of the optimum were prepared with catalyst lengths of 11.25cm (base:catalyst ratio of 7:1.25) and 6.75 cm (base:catalyst ratio of 7:0.75), mixed in accordance with the procedure outlined above and the mean working time was determined with the penetrometer assembly (n=5). The extrusion-mixed material was placed into the mixing machine (Pentamix™ 2; 3M ESPE, Seefeld, Germany) and a special mixing tip attached before the mixing process commenced. Once the material was mixed it was then placed into the cylindrical mould in accordance with the procedure outlined above and the mean working time was determined with the penetrometer assembly.

Elastic Properties

To assess the elastic properties of the hand-mixed and extrusion-mixed polyether impression materials, five specimens were prepared for each hand-mixed and extrusion-mixed regime investigated.

Elastic Recovery

The hand-mixed polyether material was laid out in two equal lines measuring 9 cm, placed onto a mixing block and mixed together with a clean spatula for 45 s. The mixed material was placed into a cylindrical mould (20 mm height and 12.5 mm diameter) and an acetate strip and glass plate were pressed on top to extrude any excess material. The mould was placed into a water-bath maintained at 35 ± 1°C for 195 s (recommended residence time in the mouth) and following removal and within 60 s the specimen was retrieved from the mould[6]. Three measurements were made on the length of the specimen using a digital micrometer accurate to 10 mm (Mitutoyo, Kawasaki, Japan) and the average length recorded. The specimen was then immediately placed onto the table of a tensile testing machine (Instron Model 5565, High Wycombe England), deformed to 6.0 ± 0.1 mm within 1 s and released slowly for 5s [6]. Three measurements were made on the length of the specimen after a further 120 s using the digital micrometer and the average length recorded[6]. The procedure was repeated five times to provide a constant measurement for the elastic recovery. The procedure was repeated for the hand-mixed polyether material manipulated with base:catalyst ratios of 7:0.75 and 7:1.25 and the extrusion-mixed polyether investigated. The elastic recovery *K* (%) was calculated using equation 1

$$K = 100 - \left[100\left(\frac{a-b}{20}\right)\right] \qquad \text{Equation 1}$$

where *a* is the original length (mm) and *b* is the deformed length - 115 s after the deforming force has been removed (mm) [6].

Strain in Compression

Five specimens were prepared to investigate the strain in compression of the hand-mixed and the extrusion-mixed polyether impression materials for each regime investigated. The hand-mixed and encapsulated polyether cylindrical specimens (20 mm height and 12.5 mm diameter) similar to those for elastic recovery were prepared. Following removal of the mould after 195 s in the water-bath maintained at 35 ± 1°C and specimen retrieval within 60 s, the specimen was immediately placed onto the table of the tensile testing machine and subjected to an initial load of 1.22 N for 30s [6]. A reading (*A*) was obtained by taking three measurements of the length of the specimen using the digital micrometer and the average length recorded[6]. The specimen was then gradually subjected to an increased load of 12.25 N over 10s which was maintained for a further 30s and reading (*B*) was obtained by taking three measurements of the length of the specimen using the digital micrometer and the average length recorded[6]. The procedure was carried out five times to provide a consistent measurement for the strain in compression of the hand-mixed polyether material at the base:catalyst ratios investigated and the extrusion-mixed polyether. The strain in compression *E* (%) was calculated using equation 2

$$E = 100\left(\frac{A-B}{20}\right)$$ Equation 2

where *A* is the original length (mm) after application of the initial load and *B* is the strained length (mm) [6].

Statistical Analysis

Multiple comparisons of the working characteristics, elastic recovery and strain in compression group means were made using a one-way analysis of variance (ANOVA). Tukey's multiple range test was employed Post Hoc at a significance level of P<0.05.

RESULTS

3.1 Working Time

The working time results in Table 1 highlighted a significant reduction in the working time (P=0.001) at a base:catalyst ratio of 7:1.25 but no significant reduction in the working time (P>0.05) at a base:catalyst ratio of 7:0.75 when the Post Hoc Tukey's multiple range test comparisons were made with hand-mixing at a base:catalyst ratio of 7:1. (Table 1). Additionally, no significant difference (P>0.05) in the working time was identified for the extrusion-mixed compared with the hand-mixed polyether at the optimum base:catalyst ratio (P<0.05).

Table 1. *The mean and standard deviations in seconds (s) for the working times of the hand-mixed and extrusion-mixed polyether impression materials. (* represents significant difference (P<0.05) in the group means of the working times examined using Tukey's multiple range test comparisons Post Hoc)*

Working Times	*Mixing Regime*	*Mean (Standard Deviation)*
Working Time	Hand-mixed (7:1)	165 (2) s
Working Time	Hand-mixed (7:1.25)	113 (2) s*
Working Time	Hand-mixed (7:0.75)	178 (1) s
Working Time	Extrusion-mixed	164 (1) s

Elastic Properties

Elastic Recovery

When the mean elastic recovery results (Table 2) were examined using Tukey's multiple range test when Post Hoc comparisons of the group means at a significance level of P<0.05, no significant differences in the elastic recovery was identified with increasing or decreasing the catalyst length by 25% compared with the optimum base:catalyst ratio. The Tukey's multiple range test Post Hoc comparisons also identified no significant difference in the group means of the elastic recovery for the extrusion-mixed polyether compared with the hand-mixed polyether impression material mixed at the optimum base:catalyst ratio.

Strain in Compression

When the group means of the strain in compression data (Table 2) was examined using Tukey's multiple range test comparisons Post Hoc, no significant difference in the strain in compression was identified (P<0.05) when increasing the catalyst length by 25%, although a significant increase in the strain in compression was identified (P<0.001) on decreasing the catalyst length by 25% compared with the optimum base:catalyst ratio Additionally, no significant difference in the strain in compression was identified for the extrusion-mixed polyether compared with the hand-mixed polyether impression material mixed the optimum base:catalyst ratio when examined using Tukey's multiple range test comparisons Post Hoc.

DISCUSSION

The mechanical manipulation of dental materials has been advocated to minimise variability in the dental clinic by members of the dental team. Rubenstein[8] assessed the mean compressive fracture strength, solubility and working characteristics of both hand-mixed and mechanically mixed silicate cement and concluded that the results favour mechanical mixing over hand-mixing due to the fast (within 20 s) consistent mixing with no wastage of powder and no need to clean a glass slab or spatula. Today, encapsulated luting cements[9-12] and restoratives[10,11,13-14] are routinely employed to eliminate variability on mixing by

Table 2. *The mean and associated standard deviations of the elastic recovery and strain in compression for the hand-mixed and extrusion-mixed polyether impression materials. (* represents significant difference (P<0.05) in the group means of the strain in compression examined using Tukey's multiple range test comparisons Post Hoc)*

Property	*Mixing Regime*	*Mean (Standard Deviation)*
Elastic Recovery	Hand-mixed (7:1)	99.2 (0.5)%
Elastic Recovery	Hand-mixed (7:1.25)	99.6 (0.3)%
Elastic Recovery	Hand-mixed (7:0.75)	98.8 (1.0)%
Elastic Recovery	Extrusion-mixed	99.1 (0.6)%
Strain in Compression	Hand-mixed (7:1)	2.7 (0.3)%
Strain in Compression	Hand-mixed (7:1.25)	3.1 (2.0)%
Strain in Compression	Hand-mixed (7:0.75)	9.1 (2.5)%*
Strain in Compression	Extrusion-mixed	3.0 (1.0)%

standardising the relative proportions of the constituents. Dental amalgam was supplied in encapsulated form in the 1970s[15] resulting in a reduction in the potential for mercury vapour contamination of the dental team as contact with liquid mercury was eliminated. More recently an extrusion-mixed polyether impression material has been introduced to the dental market which enables mixing in seconds with none of the mess associated with hand-mixing polyether impression materials. In addition the pre-measured proportions of the relevant constituents eliminate the potential for variability in proportioning the materials.[9-14]

Working Time

Increasing the catalyst volume would be expected to introduce more aromatic sulfonic acid ester into the mixture thereby causing more cross-linking to occur with the ethylene imine terminal groups of the base paste[1]. It is possible that the increased catalyst volume may have resulted in the significant decrease in the working time compared with mixing at the optimum base:catalyst ratio. Conversely decreasing the catalyst volume by 25% would have expected to yield the opposite effect namely, causing less cross-linking to occur within the impression material, and thereby increasing the working time compared with the optimum base:catalyst ratio which was not evident in the results. In an attempt to explain the disparity in the results an examination of the dental literature was undertaken. Skinner and Cooper[16] were the first investigators to attempt to assess the setting characteristics (working and setting times) of polysulfide rubber impression materials with the aid of a penetrometer. A standard Gillmore needle was applied to the surface for 10 s with the test repeated every 30 s. As long as penetration of the material occurred, the material was still considered to be workable. The setting time was defined by Bovis et al.[17] as "the number of minutes elapsed from the starting of the mix to the time when the needle fails to make a perceptible circle on the surface of the specimen when allowed to rest thereon under its own weight." A multitude of penetrometer test methods have evolved for the assessment of polysulfide[16,18] and silicone[19-20] impression materials because of the possible combinations of sample size, temperature of test, time the needle is applied for along with the size and weight of the penetrometer needle. Unfortunately, the penetrometer test results were found to be extremely operator dependent and the test method required careful and skillful attention and as a result proved to be expensive in operator time[17]. It is suggested that despite the 25% variance in the catalyst length chosen in the current study, the test was too operator dependent to enable accurate quantification of the working time. In ISO 4823:2000[6] a linear variable displacement transducer (LVDT) is recommended to assess the working time, however, a LVDT was not available and therefore the available penetrometer test assembly was employed.

The extrusion-mixed polyether material had an identical working time (164s) to the hand-mixed material (165s) indicating that no significant advantage of mechanical mixing. If both extrusion-mixed and hand-mixed polyether impression materials were exactly the same composition the extrusion-mixed material would possibly have been expected to have a shorter working time due to the extra energy introduced into the system during mechanical mixing[21] compared with hand-mixing. However it is suggested that the modification of the base:catalyst ratio by the manufacturers to control the working time under the mechanical mixing conditions was responsible for the non significant differences reported between the extrusion-mixed and hand-mixed polyether impression materials investigated.

Elastic Properties

The mechanical properties that determine the success or failure of polyether impression materials in accordance with the specification standard are elastic recovery and strain in compression (ISO 4823:2000[6]). The ideal polyether impression material should have 'bouncebackability'[22] namely, have 100% elastic recovery and negligible dimensional change (0% strain in compression), following removal from the mouth. Permanent strain will occur with elastomeric materials and the aim is to optimise the 'bouncebackability' defined as "the ability to be successful again after a period of failure" [22]. The more rapidly the material is loaded and unloaded in the oral environment the more elastically it will respond[1]. The elastic recovery and strain in compression tests were carried out to mimic the clinical situation by loading and unloading the polyether material. The set impression should also have sufficient flexibility to be withdrawn from undercuts and elastic enough to recover without under going permanent strain during removal.

Intuitively, one would expect that increasing (or decreasing) the catalyst volume by 25% would result in more (or less), cross-linking to occur between the aromatic sulfonic acid ester of the catalyst paste and the ethylene imine terminal groups of the base paste[1]. One would therefore have expected an increase (or decrease), in the mean elastic recovery results compared with mixing at the optimum base:catalyst ratio. However, the mean elastic recovery results were not significant ($P>0.05$) from the optimum base:catalyst ratio with ±25% catalyst lengths. Similarly, increasing (or decreasing) the catalyst length by 25% would be expected increase (or decrease) in the mean strain in compression. Reducing the catalyst length significantly increased the mean strain in compression ($P=0.001$) although increasing the catalyst volume did not ($P>0.05$) when compared with the optimum base:catalyst ratio.

The elastic properties (elastic recovery and strain in compression) for the extrusion-mixed polyether impression material were similar to those recorded for hand-mixed polyethers mixed at the optimum base:catalyst ratio. It has been widely reported that mechanical mixing introduces porosity into encapsulated luting cements and restoratives[9-14] but the homogeneous mixing of the constituents[12] counteract this porosity phenomenon resulting in similar mechanical properties. The authors suggest that extrusion-mixed polyether impression material has no significant advantage over hand-mixed polyethers mixed at the optimum base:catalyst ratio.

CONCLUSION

Hand-mixing polyether impression materials with 25% more catalyst than that recommended significantly decreased the working time while hand-mixing with 25% less catalyst than that recommended significantly increased the strain in compression. The extrusion-mixed polyether impression material provided similar working time, elastic

recovery and strain in compression to the hand-mixed polyether mixed at the optimum base:catalyst ratio.

ADDRESS FOR CORRESPONDENCE

Garry JP Fleming, Materials Science Unit, Division of Oral Biosciences, Dublin Dental School & Hospital, Trinity College Dublin, Dublin 2, Republic of Ireland. E-Mail: garry.fleming@dental.tcd.ie

REFERENCES

1. Van Noort R. Impression Materials. In: Van Noort R, editor. Introduction to Dental Materials. 3rd ed. Oxford: Mosby; 2007. p.186-208.
2. McCabe JF, Walls AWG. Requirement of direct filling materials and historical perspectives. In: McCabe JF, Walls AWG editors. Applied Dental Materials 8th ed. Oxford: Blackwell; 1998. p.154-6.
3. Wassell RW, Baker D, Walls AWG. Crowns and extra-coronal restorations: impression Material and Technique. *British Dental Journal*, 2002; **192:** 679-84, 687-90.
4. Pratten DH, Craig RG. Wettability of a hydrophilic addition silicone. *Journal of Prosthetic Dentistry* 1989; **61**: 197-202.
5. Vassilakos N, Fernandes CP, Nilner K. Effect of plasma treatment on the wettability of elastomeric impression materials. *Journal of Prosthetic Dentistry* 1993; **70**: 165-71.
6. ISO 4823:2000. International Standard for Dentistry - Elastomeric Impression Materials.
7. ISO 1563:1990(E). International Standard for Dental Alginate Impression Material.
8. Rubenstein J. Silicate cements prepared with mechanical mixers. *Journal of the American Dental Association* 1962; **65**: 310-6.
9. Fleming G.J.P, Shortall A.C.C, Shelton R.M, Marquis P.M. Encapsulated versus hand-mixed zinc phosphate dental cement. *Biomaterials* 1999; **20:** 2147-53
10. Nomoto R, McCabe JF. Effect of mixing methods on the compressive strength of glass ionomer cements. *Journal of Dentistry* 2001; **29**: 205-10.
11. Nomoto R, Komoriyama M, McCabe JF, Hirano S. Effect of mixing method on the porosity of encapsulated glass ionomer cement. *Dental Materials* 2004; **20**: 972-8.
12. Prentice LH, Tyas MJ, Burrow MF. The effect of mixing time on the handling and compressive strength of an encapsulated glass-ionomer cement. *Dental Materials* 2005; **21**: 704-8.
13. Fleming GJP, Zala DM. An assessment of encapsulated versus hand-mixed glass ionomer restoratives. *Operative Dentistry* 2003; **28**: 168-77.
14. Fleming GJP, Kenny SM, Barralet JE. The optimisation of the initial viscosity of an encapsulated glass-ionomer restorative following different mechanical mixing regimes. *Journal of Dentistry* 2006; **34**: 155-63.
15. The change to encapsulated amalgam. *New Zealand School Dental Service Gazette* 1976; **37**: 9-10
16. Skinner EW, Cooper EN. Desirable properties and the use of rubber impression materials. *Journal of the American Dental Association* 1955; **51**: 523-36.
17. Bovis SC, Harrington E, Wilson HJ. Setting characteristics of composite filling materials. *British Dental Journal* 1971; **131**: 352-6.
18. Miller WAC, Hansen WC, Dickson G, Sweeney WT. Physical properties of synthetic-rubber-base dental impression materials. *Journal of the American Dental Association* 1960; **60**: 211-23.
19. McLean JW. Physical properties influencing the accuracy of silicone and Thiokol impression materials. *British Dental Journal* 1961; **110**: 85-91
20. Miller N, Myers GE. Silicone impression materials. *Journal of Prosthetic Dentistry* 1962; **12**: 951-61.
21. Bass EV, Wing G. The mixing of encapsulated glass ionomer cement restorative materials. *Australian Dental Journal* 1988; **33**: 243.
22. Word invented by ex-player and now ex-manager of Crystal Palace FC Iain Dowie. Personal Communication 2004 to Sky Sports 1. Definition: The ability to be successful again after a period of failure.