# IMMEDIATE POLYMERIZATION SHRINKAGE IN LIGHT CURED RESTORATIVE RESINS

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#### ABSTRACT

The purpose of this study was to evaluate the immediate polymerization shrinkage of restorative resins following activation with different amounts of light energy.

The composites used were: Z-100 (3M-ESPE), Z-250 (3M-ESPE), Ariston pHc (Ivoclar-Vivadent), Filtek P-60 (3M-ESPE), SureFil (Dentsply), Tetric Flow (Ivoclar-Vivadent), Silux Plus (3M-ESPE) and F-2000 (3M-ESPE). The percentage shrinkage after an 80-second activation was determined by means of a capillary mercury dilatometer. The power of the light beam that reached the unpolymerized material was regulated at 350 mW/cm² (Group I) or 275 mW/cm² (Group II) positioning the end of the light source (XL-3000, 3M-ESPE) either in contact with or 5 mm from the dilatometer stopper. Four measurements were taken for each material and experi-

mental condition, reading the dilatometer scale one minute after light activation. Data were recorded as percentage volumetric shrinkage as a function of the material volume calculated from the specimen mass and density. Analysis of variance and Tukey's test were used for statistical evaluation. Shrinkage was found to be significantly greater in Group I-1.43% (0.43) - than in Group II-1.04% (0.38). Some significant differences were found among some of the products.

A reduction in polymerization shrinkage occurred concomitantly with the reduction in light activation energy and varied according to the organic content and the physical and chemical characteristics of each material.

**Key words:** Polymerization, shrinkage, dilatometer, restorative resins.

# CONTRACCIÓN DE POLIMERIZACIÓN INMEDIATA EN RESINAS RESTAURADORAS FOTOCURABLES

#### RESUMEN

El objetivo de este estudio fue evaluar la contracción de polimerización inmediata en resinas restauradoras al ser activadas con diferentes cantidades de energía lumínica.

Los composites utilizados fueron: Z-100 (3M-ESPE), Z-250 (3M-ESPE), Ariston pHc (Ivoclar-Vivadent), Filtek P-60 (3M-ESPE), SureFil (Dentsply), Tetric Flow (Ivoclar-Vivadent), Silux Plus (3M-ESPE) y F-2000 (3M-ESPE).

Luego de una fotoactivación de 80 segundos, se determinó el porcentaje de contracción con un dilatómetro de mercurio. La potencia del haz de luz que recibió cada material fue regulada en 350 mW/cm² (Grupo I) ó 275 mW/cm² (Grupo II) posicionando, respectivamente, el extremo de la fuente lumínica (XL-3000, 3M-ESPE) en contacto ó a 5 mm del tapón del dilatómetro.

Se realizaron cuatro determinaciones para cada material y situación experimental, leyendo la escala del dilatómetro luego de un minuto de concluida la fotoactivación.

Se registraron los porcentajes de contracción volumétrica en función del volumen del material calculado a partir de la masa y densidad de cada probeta. Para la evaluación estadística de los resultados se empleó análisis de variancia y prueba de Tukey.

Se encontró una contracción significativamente mayor en el Grupo I - 1,43 % (0,43) que en el Grupo II -1,04 % (0,38). Se hallaron diferencias estadísticamente significativas entre algunos materiales.

Se produjo una reducción en la contracción de polimerización al disminuir la energía de activación lumínica y ésta varió en función del contenido orgánico y las características fisicoquímicas de cada composite en particular.

Palabras clave: Polimerización, contracción, dilatómetro, resinas restauradoras.

## INTRODUCTION

Whenever a convenient light energy is applied to a photo-cured resin, the material is converted to a "rigid" solid after going through a viscoplastic stage. At the same time, shrinkage can be observed by comparing the initial and final volumes (1).

Immediately after the material is no longer a viscous body that can flow accommodating its shape to the dimensional changes that result from the polymerization reaction, it is considered to be in the above-mentioned viscoplastic or gel state (2).

Several procedures can be used to measure the polymerization shrinkage of restorative resins. The mercury or water dilatometer, the difference in density and the disk deflection methods are the most frequently mentioned in the literature (3-10).

The amount of shrinkage is cited to be from 4 to 9 percent in volume for unfilled resins and from 1 to 5% v/v for composites and resin modified cements; post-gelation shrinkage ranges from 0.2 and 2% (2). The amount of contraction is related to the combination of monomers that are involved, their molecular weight and the degree of polymerization, and the conversion that is obtained (11).

As a consequence of the contraction and the stresses it generates, adhesive failures (marginal leakage) or cohesive failures, such as cracks produced within the structures that are involved in the adhesive procedure (e.g., tooth and resin), can be produced (11). A reduction in the light intensity could increase the duration of the pre-gel state and allow for a partial relaxation of those stresses and afford greater possibilities of obtaining marginal integrity in restorations (2, 12). It has also been mentioned that lower rigidity (lower elastic modulus) in the resin also allows for better stress relief and marginal seal (2,13).

Even when restorative resins exhibit different polymerization shrinkage, marginal integrity is mainly determined by the stress that is developed in the adhesive interface (1,12,13). Such a behavior is not only a function of the volumetric contraction but also of a complex interaction between the volumetric contraction and cavity preparation configuration, intensity of light curing and viscoelastic behavior of the restorative material (1, 14-16).

The objective of this study was to evaluate the immediate volumetric shrinkage of several restorative resins when polymerized with variable light energy.

### MATERIALS AND METHODS

The products that are listed in table 1 were used in this study because they are representative of resins with different amounts of filler, different consistencies and different handling characteristics.

Their density was gravimetrically determined with four samples for each material (approximately 100µl in volume) using a mercury picnometer. A mercury dilatometer was used for volumetric shrinkage determinations (Fig. 1).

Two groups (Groups I and II) of four determinations were carried out with each of the products. A volume of approximately 100 µl of unpolymerized material was dispensed on the glass stopper base, which was then put in place to obtain a tight seal in the device. An initial reading of the height of the mercury column was immediately taken and polymerization was then activated for 80 seconds with the light of a QTH unit (XL-3000, 3M-ESPE) with a 7 mm diameter end. The height of the mercury column was recorded again 40 and 80 seconds after initiating light exposure and one minute after interrupting it.

In Group I, the power output that reached the material was regulated at 350 mW/cm² while in Group II it was set at 275 mW/cm². These values were obtained by placing the active end of the curing unit either in contact with or 5 mm from the dilatometer stopper, respectively. This distance was obtained with a steel cylinder that was attached to the end of the unit.

TABLE I. Products used in this study.					
MATERIAL	MANUFACTURER	BATCH			
Z 100	3M-ESPE	8004			
FILTEK Z 250	3M-ESPE	9 AT			
ARISTON pHc	IVOCLAR VIVADENT	A 05619			
FILTEK P60	3M-ESPE	9 AF			
SUREFIL	DENTSPLY	980715			
TETRIC FLOW	IVOCLAR VIVADENT	A 0 1125			
SILUX PLUS	3M-ESPE	7 CH			
F 2000	3M-ESPE	8 AY			

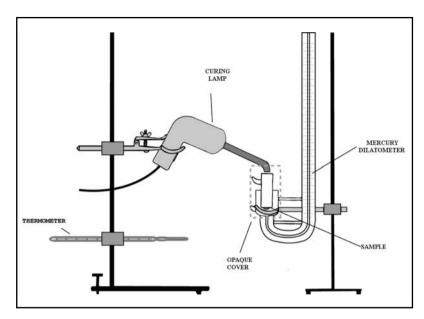


Fig. 1. Device used for volumetric shrinkage determinations.

The specimen was then removed from the dilatometer and weighed to the nearest 0.1 mg with an analytical scale (Mettler, type H6). The volumetric shrinkage was established from the difference in readings of the height in the mercury column multiplied by the capillary tube section area (0.13 mm<sup>2</sup>). The percentage of volumetric shrinkage was calculated in terms of the ratio between total volumetric shrinkage and the specimen volume calculated from its weight and the density value for the specific material.

Analysis of variance was performed (P<0.01) for both factors and their interaction.

Tukey's test was used for statistical evaluation. Significance level was established at P<0.05.

#### RESULTS

Arithmetic means of density values (g/cm³) for each material were: Filtek **P-60**: 2.20 – **Z-100**: 2.19 -

SureFil: 2.11- F 2000: 2.08 – Tetric Flow: 2.04- Filtek Z-250: 1.97- Ariston pHc: 1.93- Silux Plus: 1.61. Table 2 summarizes the analysis of variance that revealed statistical significance (P<0.05) for both factors and their interaction.

Immediate (one minute after finishing activation) shrinkage results under the two experimental conditions are shown in table 3. In Group I, similar values were obtained in resins with similar amounts of filler. Tetric Flow exhibited the highest shrinkage value and SureFil showed the smallest value, followed by Silux Plus. Values were significantly lower in Group II than in Group I, but with a similar ranking of products.

#### **DISCUSSION**

When a composite resin polymerizes, we can see a shrinkage or contraction. This contraction is a product of the diminished distance between the monomers. At the liquid state, they are related by hydrogen bonds and van der Walls forces (distances around 0.3-0.4 nm), but in the solid they are related by covalent bonds, with length of about 0.15 nm (5). In the dental literature, shrinkage is a densification within a polymeric system with no appreciable loss of mass and it is non-directional (i.e. scalar). However, shrinkage causes flow or deformation with a clear direction, depending on the shrinkage distribution, the mechanical properties, and the boundary conditions (5,10). Shrinkage patterns are the result of a dynamic a complex of factors, especially, the gel-point, the development of shrinkage and the Young's modulus as function of time, and the degree of cure. In light-curing resin compos-

TABLE II. Analysis of variance.							
FACTOR	df	SS	MS	F	Р		
DISTANCE (A)	1	2.37	2.37	363.75	<0.01		
MATERIAL (B)	7	9.40	1.34	206.45	<0.01		
INTERACTION A*B	7	0.21	0.03	4.59	<0.01		
RESIDUAL	48	0.31	0.01				
TOTAL	63	12.28					

TABLE III. Immediate shrinkage for group I and group II.						
MATERIAL	GROUP I		GROUP II			
	MEAN	SD	MEAN	SD		
T. Flow	2.35%	0.03%	1.95%	0.07%		
Z 100	1.68%	0.03%	1.12% a	0.03%		
F 2000	1.53% a	0.02%	1.04% a,b	0.02%		
Ariston pHc	1.47% a	0.01%	0.98% a,b	0.29%		
Filtek Z 250	1.20% b	0.03%	0.92% a,b,c	0.04%		
Filtek P 60	1.16% b	0.04%	0.85% b,c	0.03%		
Silux Plus	1.04% c	0.03%	0.70% c	0.04%		
SureFil	1.03% c	0.06%	0.83% b,c	0.02%		

Note: Means identified with the same letter within each group are not significantly different (P>0.05).

ites, these factors are driven by the curing light intensity (5).

Since the introduction of composites as dental restorative materials, the significance of boundary conditions determined by the fact that the material polymerizes inside a cavity with a specific shape, has been acknowledged as a reason for composites not to shrink uniformly in every direction (15). Hence, the term "effective shrinkage" is used for the phenomenon that is observed when composite shrinkage is restricted by cavity walls in clinical situations. It is opposed to "free shrinkage" for a composite sample which is allowed to shrink freely in an experimental set-up (e.g. dilatometer) (4,5,15,16).

When dental resins are applied using direct techniques and cured in situ, the setting stresses that develop tend to be transferred to the tooth-restoration structure, thus threatening its integrity and the seal between its components. Hence, polymerization shrinkage can be expected to have negative effects on the performance of resin based filling materials, luting cements, bonding agents, fissure sealants, etc. (15).

Factors such as mechanical and physical properties, polymerization shrinkage, and configuration of the cavity preparation and application techniques have to be considered for the selection of a restorative material in a specific clinical situation (1,14).

The contraction values that were found are evidence of a somewhat direct relationship between these values and the organic content of a restorative resin under the experimental conditions. Tetric Flow, the product with the highest organic content, showed the highest shrinkage values.

On the other end, the values for the condensable and microfilled products were found to be the lowest. The former has a high inorganic filler load, while in the latter pre-polymerized particles are included in the formulation. Neither of these components polymerizes under the curing light and so no contribution to the contraction value can be expected from them.

A reduction in the energy power to which the resins were exposed was found to lead to a statistically significant reduction in contraction values.

Such a difference could be associated with a lower final degree of polymerization of the resin in Group II (12,15,16). If such were the case, a similar reduction in mechanical properties, such as a lower elastic modulus could be expected (1). Both situations favor marginal quality of restorations due to lower stresses in the interfaces (1,14,15).

From this point of view, the use of low intensity light emission during restorative procedures should be considered. However, the influence of such a technique on the final properties of the different products has to be studied.

It can be concluded that:

- The amount of volumetric shrinkage was related to the amount of the polymerizing organic fraction in each specific product.
- A reduction in the activating energy acting without a modification of curing times led to an overall reduction in the final volumetric shrinkage percentage.

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