Porosity of resin cements and resin-modified glass-ionomers

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Abstract: ***Purpose:*** To quantify the internal free surface in various resin cements and glass-ionomer-based materials. ***Materials and Methods:*** Materials tested were Nexus fluid (NXF) and viscous (NXV), Vitremer (VTM), Fuji II LC (FII), Vitremer Luting Cement (VLC), Dyract (DYR) and Compoglass (COM). Samples (n=5) were made of each material between two microscopic glass slides under same weight. With a transmitted light microscopy, four zones of each sample were evaluated, finding the number of porosities per mm^2 (NP), the average radius of porosities (RP), the ratio of total area of surface porosities (μ^2) to area (mm^2) of specimen surface (TA) and the ratio of total volume of porosities (μ^3) to area (mm^2) of material surface (VP). ***Results:*** Median test was used. NP: NXV, NXF and DYR had smaller NP than VTM and FII and (likely) than VLC ($P > 0.0000001$). RP was smaller for DYR than for VLC and (likely) than for NXV ($P = 0.00019$). TA: NXV, NXF and DYR had smaller TA than VTM, FII and VLC ($P < 0.0000001$). VP: NXV and DYR had smaller VP than FII and (likely) than VTM and VLC ($P < 0.0000001$). (*Am J Dent* 2001;14: 17-21).

Clinical significance: Though it has been demonstrated that the internal porosity is directly related with contraction stress, it has not been measured yet in clinical situations, nor its influence on other clinical parameters established. There were significant differences in the content of pores in the materials studied; the ratio of total area of porosities (μm^2) to area (mm^2) of specimen surface (TA) was higher for resin-modified glass-ionomer materials (VTM, FII and VLC). The direct clinical relevance of these findings is unknown, although they highlight probable important consequences of the material's short- and long-tem behavior.

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Introduction

Resin-based cements are widely used for the cementation of esthetic inlays, onlays and veneers.[1] These materials are a mix of monofunctional monomers with a variable amount (55-70% w/w) of filler having many possible sizes, forms and compositions. Its amount is reduced in relation to restorative resins, in order to decrease viscosity and allow better adaptation of the rigid restoration to cavity.[1,2] Among its advantages are: color, adhesion to dental tissues and other materials, lesser solubility, improved mechanical characteristics and the possibility of light or dual cure.[3-5] But they have some disadvantages, such as their polymerization contraction.[2]

Resin-modified glass-ionomers are very popular materials due to their easy handling and acceptable tooth color matching, used as cements or as restorative materials. As resin is a part of the material, they also show polymerization contraction. Such curing contraction generates stress in the material, the tooth and/or the interface,[6] and if it is not properly resisted or counteracted will lead to a failure in the restoration. Some phenomena that help minimize this stress are: tooth deformation,[7] hygroscopic expansion[8] and deformation of the resin towards the adhesive surface(s) during the plastic stage of curing. This deformation, also known as "flow", is possible due to "free" or non-adhered surfaces,[9] and allows the resin mass to adapt to the new volume. Degree of flow depends upon characteristics of the material, environment and the relation of adhered to non-adhered surfaces. This ratio is also known as "configuration factor" (C),[10,11] and is defined by the formula C= adhered surface/free surface.

To facilitate the flow and relaxation of tensions, the free surface should be as large as possible, always within clinical limitations. The free surface of a restoration is the sum of the free external surface (the one that is not in contact with any adhesive surface) and the free internal surface (the one created by the internal porosity of the material). While the amount of external free surface is determined by clinical requirements, in other words, by the cavity configuration, and is quickly blocked by light curing,[12] the free internal surface is not limited by the configuration of the space that accommodates the material; it is uniformly distributed along the mass of the cement and is blocked later because of its distance from the light. Besides, because of the presence of oxygen in the porosities, their effective diameter is larger, as the curing of the most internal layer will be inhibited. In this way, the internal free surface would help in reducing stress by increasing flow in the initial critical stages.

These ideas were proposed by Alster *et al*[13] when they used experimental resins with different amounts of artificial porosities, determining the area that the porosities each took in mm^3 of resin, and studying the relation between porosity and stress. They concluded that there was a direct relation between the area of porosities and the stress during polymerization; therefore the presence of such porosities is beneficial for stress reduction.

However, it is also important to note that porosities probably deteriorate the mechanical properties, lowers the fatigue resistance of the material, accelerates the wear, facilitates superficial discoloration and causes gingival inflammation *via* plaque accumulation.[14-16]

Table 1. Materials used.

Material	Abbreviation
Nexus[a] (fluid and viscous)	NX (F & V)
Vitremer[b]	VTM
Fuji II LC[c]	FII
Vitremer Luting Cement[b]	VLC
Dyract[d]	DYR
Compoglass[e]	COM

The porosities present in some materials, namely resin cements and resin-modified glass-ionomers restorative materials and cements, were identified and compared.

Materials and Methods

Specimen preparation - One resin cement in its two viscosities and five materials (restorative and luting) based on glass-ionomer and resin were used in the study (Table 1):

Following the manufacturers' instructions carefully, one operator prepared five samples of each material and placed them over microscopic glass slides. A glass microscopic cover was placed over each, under a constant weight of 1 Kg.

When needed, curing was carried out with a lamp (Translux CL[f]) at a distance of 0 mm (in contact with the glass cover) during the time specified by the manufacturer. With a transmitted light microscope (Laborlux D[g]), a camera[g] with Ektachrome 100[h] ASA film, a histologic calibrator[g] (10 µm each interval) and a blue filter (C-12[i]), four rectangular micro-photographic slides (three peripheral and one central) of each sample were obtained (Fig. 1). This distribution was used because in preliminary laboratory work, different degrees of porosity between peripheral and central areas were detected. Areas 1 to 3 were separated 120° from each other and as close to outer bonds of specimen disk as possible. Area 4 was central. Slides were projected, and the number and diameter of all porosities for each sample were hand counted and measured.

Parameters studied - Parameters to be studied were the number of porosities per mm^2 of surface of material (NP), the average radius (µm) of porosities (RP), the ratio of total area of surface of porosities (µm^2) to area (mm^2) of specimen surface (TA) and the ratio of total volume of porosities (µm^3) to area (mm^2) of specimen surface (VP).

Data analysis - Normality (Shapiro-Wilk) and homogeneity of variance (Levene) tests were applied to all parameters because both assumptions (normality and homocedasticity) are critical if data are to be explored with parametric tests.

Distribution of porosities in fields studied in same materials – The Kruskal-Wallis test was applied on all materials to determine porosity distribution within the same specimen.

Comparison between materials - Because not all data followed a normal distribution and the Levene homocedasticity test determined that variance differed significantly between groups, median tests were applied to determine any statistically significant difference in the distribution of data around the general medians of all four parameters.

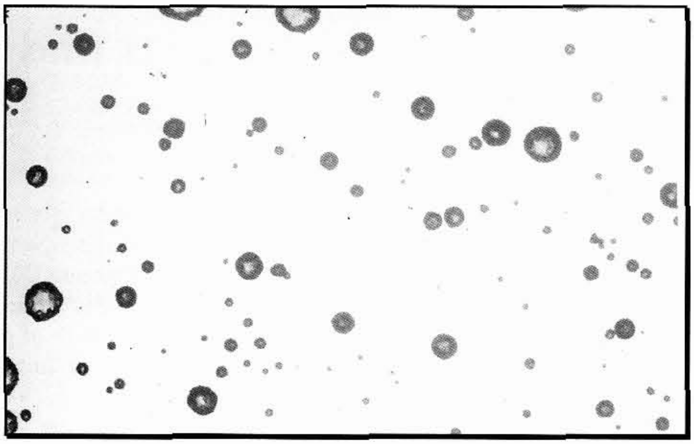

Fig. 1. Vitremer sample (x100).

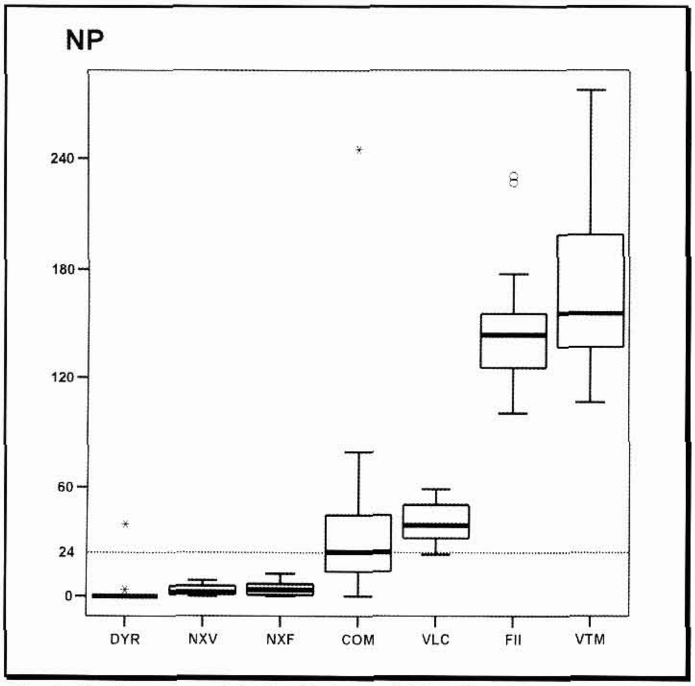

Fig. 2. Number of porosities/mm^2 of materials' surface (NP).

Results

Parameters studied - Results are shown in Table 2 and Fig. 2 (NP), Fig. 3 (RP), Fig 4 (TA) and Fig. 5 (VP). In all figures, the general median of pooled data is shown for reference.

Data analysis - Levene's test for homogeneity of variance showed that, in all parameters, the variance differed significantly ($P < 0.0001$) between groups. Shapiro-Wilk's normality tests showed that some materials' data did not follow normal distributions (Table 3).

Distribution of porosities in fields studied - Kruskal-Wallis nonparametric tests showed that only in FII material was there a statistically significant difference ($P = 0.02$) in the parameter studied among fields of the same specimen.

Comparison between materials - Results of median tests are shown in Table 4. For all parameters, distribution of data of different materials was not symmetric for both sides of the median. Significance is $P = 0.00019$ for RP and $P < 0.0000001$ for all other parameters.

Table 2. Test results.

	RP (μm)				NP				TA (μ^2/mm^2) (x10^3)				VP(μ^3/mm^2) (x10^3)			
	m	md	min	max	m	md	min	max	m	md	min	max	m	md	min	max
COM	10.7	9.8	0	25.0	39.6	24.3	0	244.8	58.6	33.6	0	28.9	284.8	100.6	0	198.2
DYR	1.4	0	0	11.7	2.4	0	0	39.8	3.8	0	0	68.2	14.4	0	0	265.2
FII	10.8	11.0	8.3	12.6	147.1	143.7	100.7	230.8	215.1	222.0	120.7	347.1	787.5	837.8	334.4	1275.4
NXF	11.6	13.9	0	22.0	4.0	3.4	0	12.3	12.8	10.5	0	38.4	73.1	37.3	0	235.6
NXV	11.8	14.0	0	23.4	3.2	2.2	0	8.8	10.3	7.7	0	60.0	59.7	42.6	0	467.5
VLC	12.6	12.2	9.8	16.9	40.0	39.0	22.8	58.9	78.4	78.3	45.7	152.8	399.9	307.4	151.8	785.3
VTM	10.6	10.7	5.9	14.0	172.7	155.7	106.7	277.3	243.4	255.5	80.9	398	899.2	941.3	159.9	1601.6

m: mean, md: median, min: minimum values, max: maximum value.

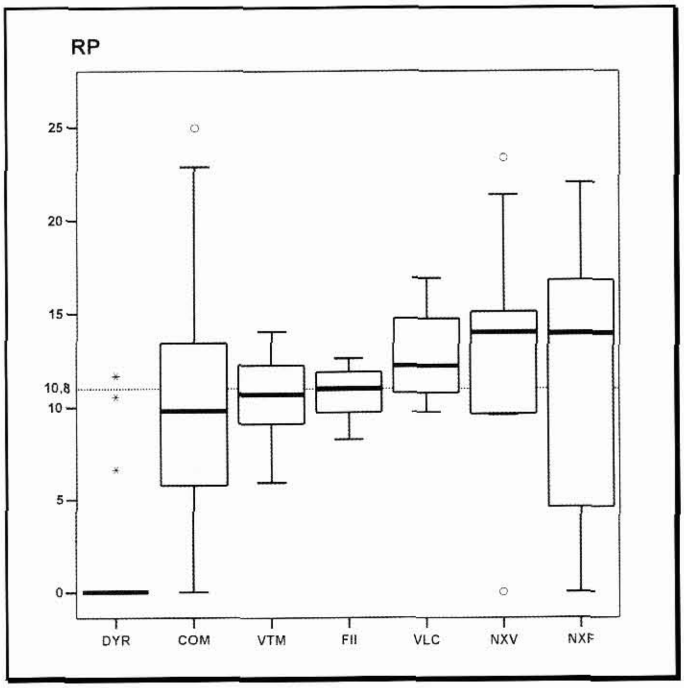

Fig. 3. Average radius (μm) of porosities (RP).

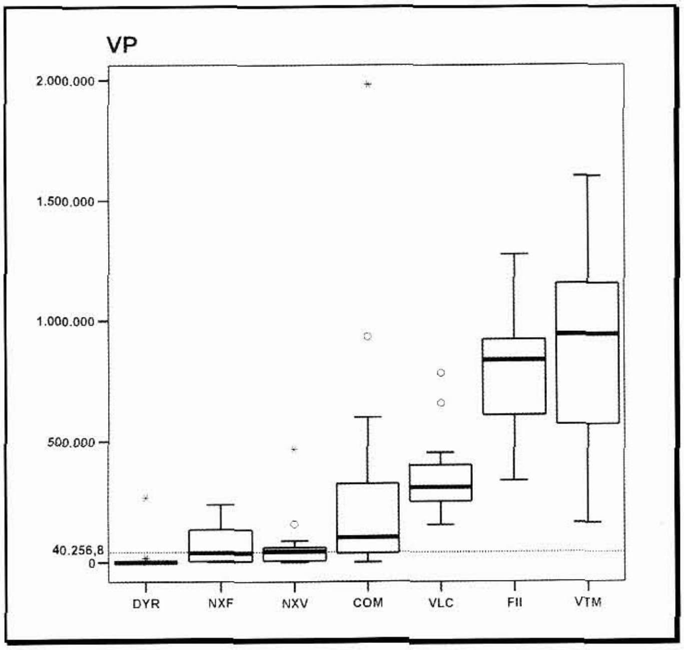

Fig. 5. Ratio of total volume (μm^3) of porosities to area (mm^2) of surface of specimen (VP).

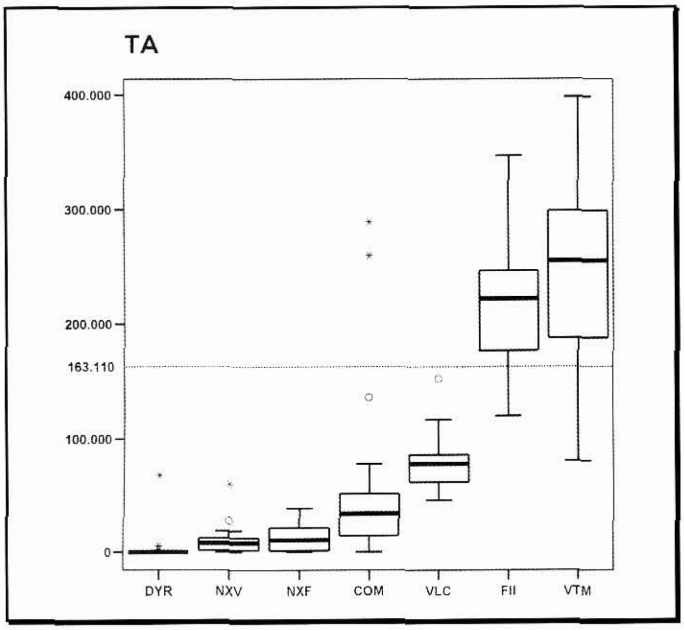

Fig. 4. Ratio of total area (μm^2) of porosities to area (mm^2) of specimen surfaces (TA).

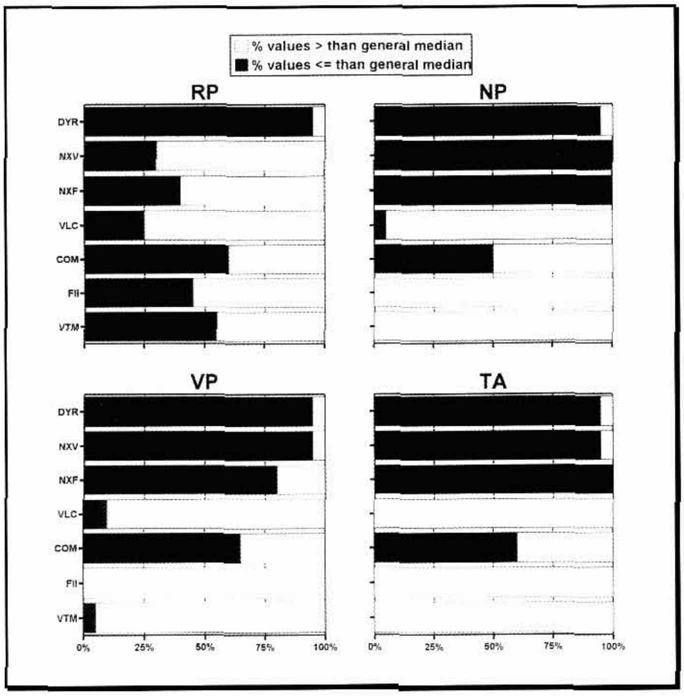

Fig. 6. Percentage of values for each material that is at both sides of the general median in the median test.

Table 3. Results of Shapiro-Wilk normality tests for all parameters.

Material	RP	NP	TA	VP
		Significance		
COM	0.235	0.010*	0.010*	0.010*
DYR	0.010*	0.010*	0.010*	0.010*
FII	0.251	0.017*	0.742	0.515
NXF	0.010*	0.124	0.020*	0.010*
NXV	0.010*	0.042*	0.010*	0.010*
VLC	0.230	0.436	0.018*	0.010*
VTM	0.772	0.021*	0.675	0.737

*differs significantly from normal distribution.

NP = number of porosities per mm^2; RP = the average radius of porosities.; TA = the ratio of total area of surface porosities (µm^2) to area (mm^2) of specimen surface; VP = the ratio of total volume of porosities (µm^3) to area (mm^2) of material surface.

These tests determined the difference in the distribution of data around the median, but did not discriminate which material was different from the others. The only way to locate differences was exploring the percentage of data of each material that was on both sides of the general median. Difference is statistically significant for both extremes of values (*i.e.* comparing NXF: 100% of cases were smaller than general median; with VTM, no cases were smaller than general median; in Fig. 6, graph corresponds to NP).

When comparing materials with values other than extremes, the difference was likely, especially considering the small P values found for all parameters.

Number of porosities per mm^2 of surface of material (NP) - From the results in Fig. 6, it is clear that NXV, NXF and DYR had smaller NP than VTM and FII and (likely) than VLC ($P< 0.0000001$).

Average radius of porosities (RP) - RP was smaller for DYR than for VLC and (likely) than for NXV ($P= 0.00019$). Ratio of total area of porosities (µm^2) to area (mm^2) of surface of material (TA). NXV, NXF and DYR had smaller TA than VTM, FII and VLC ($P< 0.0000001$).

Ratio of total volume (µm^3) to area (mm^2) of surface of material - (VP) NXV and DYR had smaller VP than FII and (likely) than VTM and VLC ($P< 0.0000001$).

Discussion

Preparation of specimens - Specimen disks were prepared with a constant weight (1 Kg), thus obtaining different thicknesses. This is the reason the parameters selected referred to area of specimen surface, instead of referring it to volume. As we used an optical surface method, only surface bubbles were measured.

Distribution of parameter in different fields of specimens - There was only one material (FII) with a significant difference in distribution of bubbles between the different fields of each specimen. We are not sure of the relevance of this single finding, but found that the porosities were generally distributed uniformly on the surface of the materials.

Comparison between materials - Median test is a nonparametric test with a low power. It can only detect if data of each material distributes uniformly (or not) around the general median. Unfortunately, the only way to discriminate between materials is to observe the median percentage distribution.

Table 4. Results of median test for all parameters.

	RP (µm)	NP	TA (µm^2/mm^2)	VP (µm^3/mm^2)
Median	10.79	24.35	163110.61	40256.76
X^2	26.4	112.4	90.4	113.2
d.f.	6			
Significance	0.00019	<0.0000001	<0.0000001	<0.0000001

X^2: Chi-*square* statistic, d.f.: degrees of freedom.

From the present results it can be stated that RP was smaller for DYR than for VLC and (likely) than for NXV ($P=0.00019$). DYR had a mean RP of 1.4 µm (range 0 to 11.7) and VLC of 12.6 (9.8 to 16.9). This means that both values were within the visible range but the one for VLC will probably have higher detrimental esthetic effect. NXV, NXF and DYR had smaller NP than VTM and FII and (likely) than VLC ($P< 0.0000001$). This will had an effect on TA: NXV, NXF and DYR had smaller TA than VTM, FII and VLC ($P< 0.0000001$), and on VP: NXV and DYR had smaller VP than FII and (likely) than VTM and VLC ($P< 0.0000001$).

Both these parameters (TA and VP) are important in predicting the behavior of materials during polymerization contraction. From this standpoint, NXV, NXF and DYR may have less stress relieving characteristics than VTM, FII and VLC.

In this study measures were carried out when the materials had set. This should cause somewhat higher results, because porosities would have dilated already, and the measurements reflected an internal free surface slightly higher than the one prior to curing. It is possible that differences must be constant before and after polymerization.

To interpret these results visually, it has to be considered that our data showed the area of porosities related to the surface area considering the whole surface of the spheres. In this way, a material such as Vitremer showed a mean rate of 243.4×10^3 µm^2 of surface of porosities/mm^2 of cement. This means that a squared mm of material will not show 0.24 mm^2 occupied by porosities, but 0.061 (Fig. 1).

The results about the internal free area are different than the ones reported by Alster *et al*,[13] probably because the materials we used were different from theirs. In addition, using a higher magnification may have enabled better detection and measurement of submicroscopic porosities.

We are not aware of any study on the most convenient, if any, free internal area of a real cementation material in order to prevent the establishment of curing stress, without detectable secondary effects on mechanical (strength, resistance to wear) or esthetic behavior. Therefore, further studies in this are needed.

a. Kerr, Orange, CA, USA.
b. 3M, St. Paul, MN, USA.
c. GC, Tokyo, Japan.
d. DeTrey/Dentsply, Konstanz, Germany.
e. Ivoclar/Vivadent, Schaan, Liechtenstein.
f. Hereaus Kulzer, Hanau, Germany.
g. Leitz Wetzlar, Germany.
h. Eastman Kodak, Rochester, NY, USA.
i. Kenco, Hong Kong, China.

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