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Porosity of different dental luting cements

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ABSTRACT

Objective. The aim of this in vitro study was to compare open porosity and pore size distribution of different types of luting cements (zinc phosphate and polycarboxylate produced by Harvard Cement, Great Britain, glass–ionomer product GC Fuji I, GC Corporation, Japan, and Panavia F, resin based composite cement, Kurraray Co. Ltd. Japan) using mercury intrusion porosimetry and use it as an additional parameter for ranging the quality of cements used in prosthetics.

Method. Samples were hand mixed in accordance with the manufacturer's instructions and formed in cylindrical test specimens. Density of samples was determined using a pycnometer while porous structure was estimated using high pressure mercury intrusion porosimeter enabling estimation of pore diameters in interval 7.5–15,000 nm.

Results. The polycarboxylate cement posses the highest porosity and specific pore volume among investigated cements. By comparison of the results obtained for zinc phosphate and glass–ionomer cement, it can be observed that according to some textural properties zinc phosphate cement is better choice (smaller specific pore volume and absence of macropores larger than 1 μm) while according to other textural properties the glass–ionomer has advantage (smaller porosity). The resin based composite cement poses the most desired porous structure for prosthetic application among the investigated cements (the lowest porosity and specific pore volume and all identified pores are smaller than 20 nm).

Significance. Based on results of this study, it is possible to estimate the efficiency of luting cements to protect the interior of tooth from penetration of oral fluids, bacteria and bacterial toxins into unprotected dentine.

1. Introduction

A number of materials are commercially available as luting cements in fixed prosthodontics. All these materials differ in chemical composition and therefore posses significantly different physical, mechanical and biological properties. Cements are usually formed by an acid/base reaction in which the acidic liquid and basic powder are combined to produce matrix of reaction products in which are embedded unreacted powder particles. In most cases, the powders are either zinc oxide or aluminosilicate glasses while the liquid are phosphoric acid, polyacrylic acid, or eugenol. In these cements, the dispersed phase, powder is having micrometric dimensions while in special group of cements, composite or resin cements the dispersed phase is having nano dimensions [1–6].

The luting cement should fulfill a great number of criteria in order to be applicable in common practice. The choice of cements is mandated to a large degree by functional and biological demands of the particular clinical situation. The

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cement with optimal performances has low viscosity and film thickness, long working time with rapid set at oral temperatures, low solubility, high compressive and tensile strengths, adhesion to tooth structure and restorative materials, anticariogenic properties, biocompatibility, etc. [1–6].

Among most commonly used the zinc phosphate, polycarboxylate, glass–ionomer and composite cements present the four distinct, characteristic groups in contemporary practice. Each of these cements according to some or even several criteria is superior to others. For instance, according to compressive strength, these cements can be ranged from highest to lowest in following order: resin; glass–ionomer; zinc phosphate; polycarboxylate. By the same order, these cements are possible to be ranged according to their solubility, but in this case from the lowest to the highest [1–6].

In this paper, the open porosity was chosen to be additional criterion for ranging luting cements. Each porous material might have three types of pores: close, through and blind pores [7]. The closed pores are not accessible to fluids. The blind pores terminate inside the material. The through pores are those that make possible the complete passageway of fluids. The open porosity includes only through and blind pores. Although porosity that includes closed pores is significant since it has a great influence on mechanical properties of a material, the importance of open porosity is in its direct impact on the possibility of penetration of undesired oral fluids, bacteria and bacterial toxins into unprotected dentine.

One of techniques that enable the measurement of open porosity is mercury intrusion porosimetry. The mercury intrusion porosimetry is method for estimation of porosity based on behavior of nonweting liquids about inundated porous object. This behavior was first described by E. W. Washburn and the governing basic equation bears his name (1) [7]:

$$D = \frac{-4\gamma \cos\theta}{P} \tag{1}$$

where γ is the surface tension, θ the contact angle, P the applied pressure and D is the pore diameter.

The equation is directly applicable with mercury, the only known liquid really suitable for porosimetry type measurements. Mercury does not enter pores spontaneously, but can be forced in to pores. Pressure required to intrude mercury in to a pore is determined by the diameter of the pore. With the increase of applied pressure the mercury is able to penetrate in to smaller pores and therefore the minimal detectable pore diameter using mercury porosimetry is the one obtained at maximal working pressure.

The IUPAC has divided pores according to their diameter in three groups: micropores ($D < 2$ nm), mesopores ($2\,\text{nm} < D < 50$ nm) and macropores ($D > 50$ nm) [8]. The mercury porosimetry enables detection from macropores dawn to larger mesopores [7].

The majority of literature provides data on porosity based on different microscopic [9–13], X-ray [14] and other techniques [15] that do not distinguish the open from closed porosity. The mercury intrusion porosimetry is seldom used for luting cement investigation [16–18] and according to our findings the comparative analysis of different cements using this technique was not performed. The ideal luting cement should be without macropores and mesopores should be reduced to minimum. Therefore, the goal of this paper was to establish the open porosity and pore size distribution of different types of luting cements (zinc phosphate, polycarboxylate, glass–ionomer and composite cement) using mercury intrusion porosimetry and use it as an additional parameter for ranging the quality of cements used in prosthetics.

2. Materials and methods

Four commercially available luting cements, each chosen to represent distinct group were used for this study. The zinc phosphate (ZnP), polycarboxylate (PC) and glass–ionomer (GI) cements were consisting of powder (A) and liquid (B), while resin based composite cement (CP) consisted of two pastes A and B. The control of composition of investigated samples was obtained by precise control of used mass of powders and pastes by means of electronic analytic balance Metter PE, Switzerland and pipette for liquids. All investigated cements were hand mixed in accordance with the manufacturer's instructions. A glass mixing pad and stainless steel spatula were used for hand mixing. The samples were obtained at room temperature $23 \pm 1\,^{\circ}$C and relative humidity $50 \pm 5\%$. After appropriate mixing time, the cements were placed into molds to produce cylindrical test specimens (6.0 ± 0.1 mm high and 8 ± 0.1 mm diameter). The mould was filled with material and the sheet of polyester film and a microscope slide was positioned on top of the material thus creating a flat surface and pressing the excess material over the brim of the mould. The microscope slide was removed and specimens were irradiated with external light source (Visilux 2, Dental Products, 3M Company, USA −450 mW/cm^2). The diameter of the light tip was 8 mm. After the initial irradiation, the specimens of composite cement were transferred to a light-curing oven (UniXS, Kulzer, Germany) for curing another 90 s. Immediately after irradiation, the specimens were removed from the mould and used in further investigation. The technical profiles of investigated cements together with mixing conditions are given in Table 1.

Density of samples was determined using a pycnometer with benzene as the displacement fluid. In order to estimate the porous structure of cements, the experiments were carried out on Carlo Erba Porosimeter 2000 using Milestone 100 Software System. This high-pressure mercury intrusion porosimeter operates in the interval 0.1–200 MPa, enabling estimation of pores in interval 7.5–15,000 nm.

3. Results and discussion

The results obtained by mercury intrusion porosimetry are presented by cumulative pore size distribution curves of investigated dental cements given in Fig. 1. Some textural properties like porosity and total pore volume per mass of sample (the specific pore volume) of investigated luting cements are calculated and shown in Table 2 together with the results obtained for pycnometer density measurements.

The given densities are the densities obtained for hardened cements. The highest density was measured for zinc phos-