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Setting characteristics and mechanical properties of self-adhesive resin luting agents

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ABSTRACT

Objective. To evaluate the degree of conversion (DC%), salt yield and mechanical properties of self-adhesive luting agents (SAA) set under dual-cure (E) and self-cure (NE) modes.

Methods. Three SAA (GC LinkAce/GCLA, MaxCem Elite/MXEL, Rely-X Unicem 2/RXUN) and an adhesive resin luting agent (Rely-X Ultimate/RXUL-control) were used. The properties tested under E and NE modes were a) DC% and phosphate salt yield after 10 min, 1 h (h) and 3 weeks (w) storage, by infrared spectroscopy; and b) the mechanical properties of 3 w-stored specimens by instrumented indentation testing (Martens hardness/HM, Elastic modulus/EIT, Elastic index/ η IT) and microscopic Vickers hardness/VH. Statistical analysis was performed by 3-way ANOVA (DC%), 2-way ANOVA (salt yield) and 1-way ANOVA (mechanical properties) at an $\alpha=0.05$.

Results. Significantly higher DC% was found in E, except from the 3 w groups of GCLA and MXEL. Within E, no significant differences were found, but within NE, there were differences in the 3 w groups of GCLA (vs 10 min) and MXEL (vs 1 h). All materials demonstrated increased salt yield in NE, with the highest values found in RXUL and RXUN. GCLA, RXUL showed the lowest HM in E and MXEL the highest in NE. The rankings of the significant differences in EIT were MXEL > GCLA, RXUN, RXUL (E) and RXUL, MXEL > GCLA, RXUN (NE), whereas for η IT RXUL, RXUN > GCLA, MXEL (E) and GCLA > RXUL > MXEL, RXUN (NE). The results of VH measurements showed an overestimation ranging from 13% up to 38% in comparison with HM.

Significance. There are significant differences in the properties tested, which may anticipate variations in the chemical, mechanical and biological performance of the products.

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1. Introduction

The developments in all-ceramic and resin composite indirect restorations have made luting procedures more demanding, aiming to integrate the restorations with hard dental tissues for enhanced strength, retention, marginal sealing and

aesthetics. Today, resin luting agents, especially the particle reinforced resin composite materials, are considered as the materials of choice, since they demonstrate superior mechanical properties [1], stability and reduced solubility [2], high

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Table 1 – The products tested and their composition.

Product shade/lot/code	Composition ^a	Manufacturer
GC LinkAce A2 shade, Lot:1211069-76 (Code: GCLA)	Resin: DUDMA, GDMA, MDP Catalysts: CHP, 2- <i>tert</i> -butyl-4,6-dimethylphenol Filler: silanated glass (50–70 wt%)	GC Corporation, Tokyo, Japan
MaxCem Elite Yellow shade, Lot: 49818224 (Code: MXEL)	Resin: HDDMA, GDMA, DUDMA, GPDMA Catalysts: TMBHP, CQ, stabilizer Filler: FAlSiO ₄ glass, SiO ₂ , Ba-glass, YF ₃ (67 wt.%)	Kerr Italia Srl, Scafati, Italy
RelyX Ultimate A1 shade, Lot:494545 (Code: RXUL)	Monomers: DCDMA, TEGDMA, substituted-DMA, GPDMA and GPDMA isomer Catalysts: sodium persulphate, sodium toluene <i>p</i> -sulphinate, TBPIN, 1-benzyl,5-phenyl barbituric acid-Ca salt Filler: silanated glass-powder, silanated SiO ₂ , oxide glass, Ca(OH) ₂ , TiO ₂ (61–71 wt%)	3M Deutschland GmbH, Neuss, Germany
RelyX Unicem 2 A2 shade, Lot: 495505 (Code: RXUN)	Monomers: TEGDMA, substituted-DMA, DCDMA, GDMA-P and GDMA isomer-P adducts Catalysts: sodium persulphate, sodium toluene <i>p</i> -sulphinate, TBPIN, cupric acetate monohydrate, 1-benzyl,5-phenyl barbituric acid-Ca salt, [(3-methoxypropyl)imino]di-2,1-ethanediyl bismethacrylate Filler: silanated glass-powder, silanated SiO ₂ , oxide glass chemicals, Ca(OH) ₂ , TiO ₂ (55–72 wt%)	3M Deutschland GmbH, Neuss, Germany

CHP: cumene hyroperoxide, CQ: camphoroquinone, DCDMA: dodecane dimethacrylate, DUDMA: diurethane dimethacrylate, GDMA: glycerol dimethacrylate, GPDMA: glycerol phosphate dimethacrylate, HDDMA: hexanediol dimethacrylate, MDP: methacryloyloxydecyl dihydrogene phosphate, TBPIN: *tert*-butyl 3,5,5 trimethyl peroxyhexanoate, TEGDMA: triethyleneglycol dimethacrylate, TMBHP: tetra methyl butyl hydroperoxide.

^a According to the safety data sheet (sds) files of the manufacturers.

adhesive strength to restorative materials and tooth structure, and improved aesthetics [3].

Resin composite luting agents have been classified into three groups depending on the adhesive system used for tooth pre-treatment [4]. Originally, 3-step adhesive systems (etchant, primer, bonding resin) had been introduced, which had been then substituted for two-step, etch & rinse (etchant, adhesive resin) and later on for two-step, self-etch (acidic adhesive primer, adhesive or bonding resin) systems, ranking the composite luting agents, accordingly. Moreover, based on the monomer chemistry and curing mechanism of the main luting agent, they were further classified as non-adhesive or adhesive and light-, self- or dual-cured. The complexity arising from these multistep application procedures, established the need for simplification of the luting protocols, to reduce chairside time, complexity and technique sensitivity [5]. The result was the design of self-adhesive composite luting agents (SAA), not requiring any means of tooth pre-treatment. The first product of this category launched in 2002 (Rely-X Unicem, 3M ESPE) as a dual-cured SAA.

The simple and user friendly luting protocol of this new agent was soon adopted by most dental materials manufacturers; a great number of SAA have been introduced in the market with many variations in composition. The main components of SAA can be classified as follows: (a) Aromatic and aliphatic dimethacrylate monomers, to form a crosslinked network, (b) acidic methacrylate monomers to adhere with enamel and dentin and copolymerize with the crosslinking monomers, (c) glass filler particles or basic compounds, to neutralize residual acidic monomers, (d) conventional silanated filler particles to provide strength by an inert reinforcing effect, (e) appropriate catalysts and stabilizers to comply with the dual-cure nature and shelf-life requirements of the materials and (f) pigments and opacifiers, to match the aesthetic requirements.

Most of the products contain sources of fluoride release, as well (fluoroaluminosilicate glass fillers, YbF₃, etc.) [4,6].

The curing capacity of these materials has been investigated in several studies. Although it has been confirmed that self-curing leads to lower degree of C=C conversion in the absence of light-curing [7–16], there is a great variation in the values obtained by various experimental designs and analytical techniques (FTIR, Raman). Moreover, despite the fact that acidic monomers are included in these materials, their neutralization capacity has been evaluated in few studies by surface pH measurements only [17–19], without any further information on the acid–base reaction involved. The mechanical properties of the SAA have been assessed, so far, by conventional methods (3-point flexural strength and modulus on bar-shaped specimens [18–20], biaxial flexural strength [13], hardness [21,22] and wear [23–25]) and were found to be similar or inferior to composite resin luting agents, provided that the SAA specimens had been light-cured, for most of the products tested.

Recently, new materials have been introduced, with improved properties and handling characteristics. The aim of the study was to comparatively evaluate the curing efficiency, examine the salt formation capacity and evaluate the mechanical properties of SAA. The null hypothesis was that there are no statistically significant differences among the products in the properties tested.

2. Materials and methods

The products used in the study are listed in Table 1. GCLA, MXEL and RXUN are SAA, whereas RXUL is a new adhesive luting agent to be used on tooth surfaces previously treated with a self-etching universal adhesive. This product, with

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