UNIVERSIDADE ESTADUAL DE CAMPINAS

FACULDADE DE ODONTOLOGIA DE PIRACICABA

EDUARDO FERNANDES DE CASTRO

ADESÃO, PROPRIEDADES MECÂNICAS E MICROESTRUTURA DE MATERIAIS CAD/CAM À BASE DE RESINA

ADHESION, MECHANICAL PROPERTIES AND MICROSTRUCTURE OF RESIN-BASED CAD/CAM MATERIALS

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Dissertação apresentada à Faculdade de Odontologia de Piracicaba da Universidade Estadual de Campinas como parte dos requisitos exigidos para a obtenção do título de Mestre em Materiais Dentários.

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Orientador: Prof. Dr. Marcelo Giannini.

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A Ata da defesa com as respectivas assinaturas dos membros encontra-se no processo de vida acadêmica do aluno.

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RESUMO

Os objetivos deste trabalho foram investigar o efeito do armazenamento em água e de tratamentos de superfícies na resistência de união por cisalhamento (RUC) de dois cimentos resinosos a materiais CAD/CAM à base de resina (RC) e avaliar as propriedades mecânicas - módulo de elasticidade (ME) e resistência flexural (RF) - dos RCs. Adicionalmente, avaliou-se a composição e microestrutura das RCs. Três RCs (Enamic, Lava Ultimate, Cerasmart) e um compósito convencional indireto (Epricord) foram testados. Para cada material, sessenta placas (14 x 7 x 1 mm) foram preparadas para o ensaio de RUC e submetidas a três diferentes tratamentos de superfície: instruções do fabricante (IF), aplicação de plasma atmosférico não-térmico (NTAP) por 30 s e NTAP + agente de união (AU). Dois cimentos resinosos foram avaliados: RelyX Ultimate (RX) e Panavia V5 (V5). Dois cilindros de cimento resinoso (1,5 mm de diâmetro x 1,5 mm de altura) foram aderidos a cada placa (n = 10), sendo um testado após 24 h de armazenamento em água destilada e o outro após um ano. Vinte barras retangulares (12 x 2 x 1 mm) de cada material indireto foram confeccionadas e submetidas ao ensaio de flexão de três pontos após 24 h ou um ano de armazenamento em água para obtenção de valores de ME e RF (n = 10). As amostras fraturadas foram examinadas em espectroscopia dispersiva de raios-X (EDS) e microscópio eletrônico de varredura (MEV). Dados de RUC foram analisados pela ANOVA quatro fatores e dados de ME e RF por ANOVA dois fatores, seguidos de teste de Tukey (α=0,05). Os grupos IF obtiveram maiores valores de RUC que os grupos NTAP e NTAP + AU para todos os materiais indiretos, cimentos resinosos e tempos de armazenamento avaliados. De forma geral, RX obteve maiores valores de RUC aos materiais indiretos que V5, com exceção de alguns grupos do Epricord (IF). Após um ano, todos grupos mostraram reduções significativas no valor de RUC, exceto alguns grupos de IF. Com relação a ME e RF, Epricord obteve as menores médias. Dentre os materiais CAD/CAM, Cerasmart obteve a menor média de ME e a maior de RF, e Enamic o maior ME e a menor RF, em ambos tempos de armazenamento. As imagens de MEV e análises de EDS revelaram que os materiais apresentam diferentes composições e microestruturas. Alguns materiais indiretos testados mostraram alterações na adesão do cimento resinoso em função dos tratamentos e tempo, o

qual também influenciou o ME e RF desses. As diferenças composicionais e estruturais foram determinantes para o comportamento das propriedades estudadas.

Palavras-chave: CAD-CAM, resinas compostas, resistência ao cisalhamento, gases em plasma, módulo de elasticidade

ABSTRACT

The purpose of this study was to investigate the effect of one-year water storage and surface treatments on shear bond strength (SBS) of two resin cements to resin-based CAD/CAM materials (RC) and evaluate the mechanical properties - elastic modulus (EM) and flexural strength (FS) - of RCs. Additionally, the compositions and microstructures of RCs were analyzed. Three RC materials (Enamic, Lava Ultimate, Cerasmart) and one conventional indirect composite (Epricord) were tested. For each material sixty plates (14 x 7 x 1 mm) were prepared for SBS test and submitted to three different surface treatments: manufacturer's instructions (MI), non-thermal atmospheric plasma (NTAP) application for 30 s and NTAP + bonding agent (BA). Two resin cements were tested: RelyX Ultimate (RX) and Panavia V5 (V5). Two resin cylinders (1.5 mm diameter x 1.5 mm height) were bonded to each plate (n = 10), one tested after 24-h in distilled water-storage and the other after one year. Twenty rectangular bars (12 x 2 x 1 mm) of each indirect material were prepared and submitted to 3-point flexural test after 24-h or one-year water storage to obtain EM and FS values (n = 10). Fractured samples were examined under energy dispersive x-ray spectroscopy (EDS) and scanning electron microscopy (SEM). SBS data were analyzed by four-way ANOVA, and EM and FS data by two-way ANOVA, followed by post-hoc Tukey's test (α=0.05). MI groups obtained higher SBS values than NTAP and NTAP + BA for all indirect materials, resin cements and storage periods tested. In general, RX displayed higher SBS means than V5, except for some groups of Epricord MI. After one year, all groups presented significant reduction of SBS, except for some MI groups. Epricord showed the lowest values of EM and FS. Among CAD/CAM materials, Cerasmart had the lowest EM and highest FS means, while Enamic had the highest EM and lowest FS means, for both storage periods. SEM images and EDS analyses showed that the RCs presented different compositions and microstructures. Some indirect materials tested showed alterations in resin cement adhesion according to surface treatment and storage time, which also influenced EM and FS. Compositional and structural differences were determinant on the performance of properties studied.

Key Words: CAD-CAM, composite resins, shear strength, non-thermal atmospheric pressure plasma, elastic modulus

SUMÁRIO

1 INTRODUÇÃO

O termo CAD/CAM (do inglês *Computer-Aided Design / Computer-Aided Manufacturing*), significa “Desenho assistido por computador / Manufatura assistida por computador”. Consiste, portanto, no uso de computadores para a confecção de desenhos tridimensionais virtuais de estruturas (CAD) a serem futuramente produzidas de forma automatizada por uma máquina guiada por computador (CAM). Esta tecnologia tem sido aprimorada e cada vez mais utilizada em diversas áreas do conhecimento devido à sua precisão, economia, reprodutibilidade e agilidade (Li *et al.,* 2015).

Os primeiros avanços da tecnologia CAD/CAM na Odontologia iniciaram na década de 80 (Liu, 2005), e desde então vem evoluindo em termos de precisão, rapidez e facilidade de uso (Albuha *et al.,* 2016). Diversas empresas tem desenvolvido *scanners*, *softwares* para desenho, fresadoras e impressoras tridimensionais (Albuha *et al.,* 2016). Paralelamente, foram desenvolvidos materiais odontológicos para restaurações indiretas que se adequem a estas tecnologias como os compósitos, resinas acrílicas, cerâmicas e materiais híbridos (Belli *et al.,* 2017; Yoshihara *et al.,* 2017).

Dentre esses materiais, os compósitos são bastante atrativos devido algumas vantagens como custo, menor desgaste das brocas de fresagem (Lebon *et al.,* 2015; Chavali *et al.,* 2017) e o fato de não necessitarem de nenhum processamento adicional como sinterização ou glazeamento, o que garante uma rápida manufatura (Awada e Nathanson, 2015). Um dos primeiros blocos de resina para CAD/CAM disponíveis comercialmente foi o Paradigm MZ100 (3M ESPE), produzido com base na composição do compósito convencional Filtek Z100 (3M ESPE) (Yoshihara *et al.,* 2017).

Atualmente, existem dois tipos de materiais CAD/CAM à base de resina (RC) comercialmente disponíveis: blocos de resina composta (Lava Ultimate [3M ESPE], Cerasmart [GC Dental Products], Shofu Block HC [Shofu], Katana Avencia [Kuraray Noritake], Brava Block [FGM], KZR-CAD H [Yamakin], Grandio blocs [Voco], Estelite Block [Tokuyama]) e uma cerâmica feldspática infiltrada por uma rede polimérica (Enamic, Vita Zahnfabrik). A diferença básica entre esses dois materiais é que o conteúdo inorgânico dos blocos de resina composta se encontra disperso na matriz resinosa, da mesma forma que nas resinas compostas utilizadas

de modo direto, enquanto o outro material, segundo seu fabricante, apresenta unidade estrutural cerâmica infiltrada por monômeros resinosos sendo classificado como material híbrido (cerâmica e compósito) (Awada e Nathanson, 2015; Yoshihara *et al.,* 2017).

As desvantagens desses materiais comparados às cerâmicas são relacionadas à adesão e propriedades mecânicas. Além disso, dependendo do tipo da formulação monomérica, eles podem possuir diferentes taxas de degradação em ambiente bucal. Essas taxas de degradação são dependentes dos diferentes níveis de sorção de água das matrizes poliméricas e degradação hidrolítica do grupamento éster dos metacrilatos (Ferracane, 2006; Van Landuyt *et al.,* 2007).

Recentemente, a 3M Oral Care dos Estados Unidos da América removeu a indicação para coroa do Lava Ultimate devido a alta taxa de descimentação (3M Notice, 2015). Os compósitos resinosos tem sido utilizados na Odontologia como materiais restauradores indiretos por mais de 35 anos (Miara, 1998) com altas taxas de sucesso e longevidade (Barabanti *et al.,* 2015). Entretanto os materiais produzidos para a tecnologia CAD/CAM ainda precisam de mais estudos que avaliem suas principais desvantagens, como reportado anteriormente. O conhecimento mais aprofundado e experiência clínica acerca desses compósitos e material híbrido podem trazer informações relacionadas aos fatores que podem levar à alta taxa de descimentação, dentre eles:

1. O alto grau de conversão monomérica dos RCs, devido às condições industriais de polimerização com pressão e temperatura controlados, resulta em melhores propriedades mecânicas devido à alta taxa de ligações cruzadas, que em contrapartida reduz o número de ligações duplas de carbono para a copolimerização com adesivos e cimentos resinosos.
2. Devido ao alto conteúdo orgânico estes materiais apresentam baixo módulo de elasticidade comparados às cerâmicas odontológicas, o que resulta em maior flexibilidade, podendo aumentar o deslocamento das coroas.

Para a cimentação dessas peças protéticas existe uma grande variedade de materiais disponíveis, entre eles cimentos convencionais, ionoméricos e resinosos. Atualmente, os cimentos resinosos são amplamente indicados e podem

variar de acordo com o modo de polimerização e mecanismo de união às estruturas dentais (Stamatacos e Simon, 2013).

A adesão de cimentos resinosos a RCs pode ser melhorada por mecanismos que aumentem a energia de superfície e molhabilidade, como o plasma atmosférico não térmico (NTAP) (Kim *et al.,* 2014). Várias pesquisas têm investigado o uso desta tecnologia buscando melhorar a adesão a diversos substratos, dentre eles: esmalte, dentina, pino de fibra de vidro e zircônia (Dong *et al.,* 2013; Hirata *et al.,* 2016). O NTAP nunca foi testado em RCs e além disso, poucos trabalhos avaliaram a resistência de união de cimentos resinosos e as propriedades mecânicas destas RCs a longo prazo (Kassotakis *et al.,* 2015; Cekic-Nagas *et al.,* 2016).

Portanto, este estudo comparou três materiais para CAD/CAM e um compósito indireto convencional analisando diferentes propriedades como: adesão (incluindo cimentos resinosos e tratamentos de superfície), propriedades mecânicas, composição e estrutura. Além disso, o efeito do tempo foi considerado neste estudo na tentativa de se predizer a longevidade clínica desses materiais, tópico esse muito explorado em pesquisa, pois muitos materiais restauradores novos apresentam boas propriedades e adequado comportamento clínico somente a curto prazo.

2 ARTIGO:

Adhesion, mechanical properties and microstructure of resin-based CAD-CAM materials

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Abstract

Statement of problem. Due to industrial fabrication method of resin-based CAD-CAM materials (RC), adhesion of resin cements to their surface is a concern. Besides, different compositions and microstructures might determine their mechanical properties and long-term clinical performance.

Purpose. The purpose of this study was to investigate the effect of one-year water storage and surface treatments on shear bond strength of two resin cements to RCs and on mechanical properties - elastic modulus (EM) and flexural strength (FS) - of RCs. Additionally, the microstructures and compositions of RCs were analyzed.

Material and Methods. Three RCs (Enamic, Lava Ultimate, Cerasmart) and one conventional indirect composite (Epricord) were tested. For each material sixty plates (14x7x1 mm) were prepared for bond strength test and submitted to three different surface treatments: manufacturer's instructions (MI), non-thermal atmospheric plasma (NTAP) application for 30 s and NTAP + bonding agent. Two resin cements were tested: RelyX Ultimate (RX) and Panavia V5 (V5). Two resin cylinders (1.5 mm diameter x 1.5 mm height) were bonded to each plate (n = 10), one tested after 24-h distilled water-storage and the other after one year. Twenty rectangular bars (12x2x1 mm) of each indirect material were prepared and submitted to 3-point flexural test after 24-h or one-year water storage to obtain EM and FS values (n = 10). Fractured samples were also examined under energy dispersive x-ray spectroscopy (EDS) and scanning electron microscopy (SEM). Bond strength data were analyzed by four-way ANOVA, and EM and FS data by two-way ANOVA, followed by post-hoc Tukey's test (α=.05).

Results. MI groups obtained higher bond strength values than NTAP and NTAP + bonding agent for all indirect materials, resin cements and storage periods tested (P<.05). In general, RX displayed higher bond strength means than V5 (P<.05), except for some groups of Epricord MI. After one year, all groups presented significant reduction of bond strength (P<.05), except for some MI groups. Epricord showed the lowest values of EM and FS (P<.05). Among CAD-CAM materials, Cerasmart had the lowest EM and highest FS means, while Enamic had the highest EM and lowest FS means, for both storage periods (P<.05). SEM images and EDS analyses showed that the RCs presented different compositions and microstructures.

Conclusions. Some indirect materials tested showed alterations in resin cement adhesion according to surface treatment and storage time, which also influenced EM and FS. Compositional and structural differences were determinant on the performance of properties studied.

Clinical Implications

For resin-based CAD-CAM materials tested manufacturers' instructions for cementation must be followed. Degradation in water may compromise adhesion and mechanical properties overtime.

Introduction

Advances on scanning, milling and printing technology have brought great expectations for a more precise, cheaper, durable and less time-consuming dentistry. The development of CAD-CAM indirect restorative materials is an example of dental technology and is available in many countries. Indirect restorations have been fabricated using composite resins, ceramics, zirconia and hybrid (ceramic/resin) materials.[1,2] The first resin block for CAD-CAM was based on the composition of a conventional direct composite resin.[2] Currently there are two types of resin-based CAD-CAM materials (RC): composite resin blocks and polymer network infiltrated feldspar ceramic (ceramic/resin hybrid). The basic difference between these materials is that composites have an inorganic content disperse within resin matrix, similar to direct composites, but with higher filler content, while the hybrid material presents an interconnected ceramic network infiltrated by a polymer.[2,3]

Some particular advantages of RCs over ceramics make these materials highly attractive for dental practice, as RCs allow less bur wear[4,5] and no need for additional post-milling processing, such as firing and glazing, therefore presenting a faster manufacturing.[3] On the other hand, adhesion and mechanical properties might change over time at a higher rate than ceramics, due to water absorption and penetration into resin matrix, which leads to hydrolytic degradation of methacrylate monomers.[6,7]

Concerns have been addressed regarding the low bond strength of resin cements to RCs and possible debonding of indirect restorations from dental preparations.[8] However, if conventional composite resins have been used in dentistry as indirect restorative materials for more than 35 years[9] with great long-term clinical success rates[10], what are the reasons for such concerns? The low number of unreacted monomers remained after polymerization available to bond with resin

cement and the elastic modulus of RCs are the main hypothetical issues. The higher degree of conversion of RCs after pressure- and temperature-activated polymerization, results in a very resistant but flexible material with few residual monomers.[1,3,11]

Adhesion of resin cements to RCs may be enhanced by mechanisms that increase surface energy and wettability, like non-thermal atmospheric plasma (NTAP).[12] This technology has been used in dentistry to improve adhesion of diverse substrates: enamel, dentine, fiber posts, and zirconia.[13,14] However, NTAP has never been tested on RCs surfaces. Besides, very few studies evaluated long-term bond strength of resin cements to RCs and mechanical properties of RCs.[15,16]

Thus, the purpose of this study was to evaluate the influence of one-year water storage and surface treatments on shear bond strength (SBS) of two resin cements to RCs and investigate mechanical properties (elastic modulus [EM] and flexural strength [FS]) of RCs. Additionally, the compositions and microstructures of RCs were also assessed. The first null hypothesis was that no significant increase in SBS would be achieved with NTAP treatment, regardless of the storage time, resin cement and indirect material. The second null hypothesis was that no significant difference in EM and FS would be obtained among indirect restorative materials, regardless of the storage time.

Material and Methods

One conventional indirect composite (Epricord, Kuraray Noritake [EP]) was used as a control and three RCs (Enamic, Vita Zahnfabrik [EN]; Lava Ultimate, 3M ESPE [LU]; Cerasmart, GC Corp. [CE]) were tested. Commercial names, manufacturers, compositions and batch numbers of all materials used in this study are listed on Table 1.

RCs were sectioned using a slow-speed diamond-wafering blade (Isomet 1000 Precision Saw; Buehler Co) in sixty slabs (14 mm x 6 mm x 1 mm), which were wet-ground with silicone carbide abrasives (Norton) up to 600-grit using a grinding machine (Automet 500; Buehler Co). Samples of conventional indirect composite (EP) were prepared using silicone (Virtual; Ivoclar Vivadent) molds and the sample dimensions were similar to RCs. After filling the molds, a light-curing unit (860 mW/cm^2 of irradiance, Valo Cordless; Ultradent) was used to polymerize the samples, which were wet-ground the same way as previously described.

Samples were submitted to three different surface treatments before resin cement application: 1. According to manufacturer's instructions of RCs and resin cements; 2. NTAP application; 3. NTAP followed by bonding agent application (BA). Two resin cements (RelyX Ultimate [3M ESPE]; Panavia V5 [Kuraray Noritake]) were placed over treated composite surfaces. Detailed adhesive cementation protocols of each experimental group are described on Table 2.

Groups that were sandblasted followed the protocol: air-abrasion with 50 μm aluminium oxide using a sandblasting unit (Microetcher II; Danville Engineering Inc.) for 10 s, 10 mm away from the surface at 60 psi. Samples were then washed and submitted to an ultrasonic bath at an ultrasonic cleanser (USC 1400; Unique) in distilled water for 5 min, followed by thorough air-drying.

NTAP equipment (Surface Plasma Tool Model SAP; Surface–Engineering and Plasma Solution) used in this study is a hand-held unit that uses argon as operating gas at a flow rate of 1 liter per minute. NTAP was applied for 30 s perpendicular to samples surface at 22°C, with a 10 mm distance between the nozzle and the samples (Fig. 1).

In order to limit the bonding area, two adhesive tapes with 1.5 mm (diameter) hole each, were prepared and placed on the surface of each sample (Fig. 2A). Following RCs treatments and bonding procedures, two silicone molds (Virtual;

Ivoclar Vivadent), each with 1.5 mm diameter and 1.5 mm height, were positioned on the tapes with their holes coinciding to those of tapes, and a resin cement filled up the orifices to form two resin cement cylinders (Fig. 2B) after light activation for 20 s, which was performed with a polywave LED light (Valo Cordless; Ultradent). Ten RCs and EP plates were prepared per group (n = 10).

Silicone molds and tapes were carefully removed and samples were stored in distilled water at 37ºC. One resin cylinder was submitted to SBS test after 24 h, and the other after one year. Prior to SBS test, specimens were dried and fixed with cyanoacrylate glue (Super Bonder; Loctite) on a testing device, which was attached to a universal testing machine (EZ-Test; Shimadzu). SBS was determined with the shear load applied by an orthodontic wire (0.08" diameter) to resin cylinder's base at a crosshead speed of 1.0 mm/min until failure. SBS values were calculated by dividing the maximum load at failure (N) by the bonding area (mm^2) and were expressed in megapascal (MPa). Bond strength data were analyzed by four-way ANOVA (indirect material, treatment, resin cement and storage time) and Tukey HSD post hoc test (α=0.05). The statistical analyses were performed by SAS for the personal computer (SAS Institute).

Tested specimens were examined with a digital microscope (KH 8700; Hirox) and representative images of different materials and failure patterns were taken at x100 magnification. Modes of failures were classified according to the following types: 1. Adhesive failure (ADE); 2. Cohesive failure within indirect resin (COR); 3. Mixed failure involving adhesive layer and indirect resin (MAR); 4. Mixed failure involving adhesive layer and resin cement (MAC); 5. Mixed failure involving adhesive layer, indirect resin and resin cement (MARC).

The same indirect restorative materials tested for SBS were used for 3-point flexural test. RCs were sectioned with a diamond-wafering blade mounted on a saw (Isomet 1000 Precision Saw; Buehler Co) under constant irrigation, into twenty rectangular bars (12 mm x 2 mm x 1 mm).[17] A silicone (Virtual; Ivoclar Vivadent) impression of one RC bar was used to obtain twenty EP samples, with the same dimensions aforementioned. Specimens were measured with a digital caliper (Starrett) to assure the exact dimensions and were stored in distilled water at 37ºC. Ten samples of each material were tested after 24 h and the other ten after one year (n = 10).

Flexural test was conducted using a universal testing machine (Instron 4411; Instron) with a 500-N load cell. Each specimen was positioned on a metal fixture with a 10-mm support span[17] and centered under loading of 1.0 mm/min crosshead speed, until fracture. EM and FS values were obtained and expressed in gigapascal (GPa) and megapascal (MPa), respectively. Data were analyzed by two-way ANOVA (indirect material and storage time) and Tukey HSD post hoc test (α=0.05). The statistical analyses were performed by SAS for the personal computer (SAS Institute).

Fractured samples of each material from flexural test were fixed in plastic stubs and sputter-coated with carbon (MED 010; Balzers Union) prior to energy-dispersive X-ray spectrometry (EDS) analysis, in order to identify chemical composition. The X-ray detector (X-Act; Oxford) was coupled to a scanning electron microscope (JSM IT 300; JEOL) and the analyses were acquired for 60 s (voltage 20.0 kV, dead time 20–30%, working distance 10 mm). For each material five repetitions were performed and images containing the identified chemical elements were obtained.

Another set of fractured samples were fixed in metallic stubs and sputter-coated with gold (SCD 050; Bal-tec), prior to scanning electron microscopy (SEM) (voltage 20.0 kV, beam width 35–60 nm, working distance 10–20 mm) observations. Micrographs of the indirect material microstructures were obtained at x250 and x1000 magnifications for EP, and x1000 and x5000 magnifications for RCs.

Results

Due to the presence of outliers, lack of normality and heterogeneity of variance, SBS data were transformed to the power of 0.6. SBS means are presented in Table 3. Resin cements applied to RCs and EP, which were treated following manufacturer's instructions, yielded the highest SBS, regardless of storage time ($P<.05$). In general, the SBS of RelyX Ultimate to RCs and EP was higher than those obtained with Panavia V5 ($P<.05$). Some exceptions were observed for regular indirect composite (EP).

At 24-h water storage NTAP treatment was either not significant different ($P>.05$) or higher than ($P<.05$) NTAP + BA, with the exception of RelyX Ultimate bonded to CE, where NTAP was lower than NTAP + BA ($P<.05$). In general, EN was the indirect material that obtained higher SBS with both resin cements following the NTAP treatment and the manufacturer's recommendations.

Regarding one-year water storage, NTAP produced significantly lower SBS than NTAP + BA for EN and CE with Panavia V5 ($P<.05$) and for CE with RelyX Ultimate. Comparing indirect materials at one year, SBS results depended on treatment and resin cement. Following manufacturer's instructions, SBS of Panavia V5 to LU showed the lowest mean ($P<.05$), but with RelyX Ultimate the same result was not found. When comparing indirect materials, applying NTAP to EP followed by Panavia V5 resulted on the highest SBS, while for RelyX Ultimate, EN e LU showed the greatest SBS means ($P<.05$). EP presented the lowest SBS when it was treated with NTAP + BA ($P<.05$), for Panavia V5. One-year storage in water reduced SBS for most of the groups ($P<.05$), except for EN bonded to Panavia V5, and EP and CE bonded to RelyX Ultimate ($P>.05$).

Representative images of each failure mode are shown in Figure 3 and failure modes distribution (in %) for all groups tested for SBS are presented in Figure 4. The most common failure mode detected was ADE, followed by COR. EP showed 100% of COR when treatment was according to manufacturers' instructions, regardless of resin cement used. EN had 46.2 - 54.5% of ADE when treatment followed manufacturers' instructions, while LU obtained 57.1 - 73.3% of ADE. MARC appeared only for EN on some groups where manufacturers' instructions were followed. For LU, NTAP+PA treatment resulted in 100% of ADE. Adhesion of RelyX

Ultimate to CE according to manufacturers' instructions showed 100% of COR, while for Panavia V5 groups, more than 80% of ADE was observed.

Concerning all indirect materials, NTAP and NTAP+PA treatments resulted in 100% of ADE failure using Panavia V5, except for EP at one-year storage, where there was 9.1% of MAR and 90.1% of ADE. For one-year evaluation, all indirect materials treated with NTAP or NTAL+PA followed by RelyX Ultimate, presented more than 90% of ADE, while the 24-h groups presented from 45.5 - 80.0% of ADE. Considerable modifications in the fracture pattern were observed when using NTAP and RelyX Ultimate resin cement, regardless of indirect material.

EM values were transformed by base-10 logarithmic, while the FS data presented normality and homogeneity of variance. EM and FS means are listed in Table 4, and stress-strain plot in Figure 5. At 24-h and one-year water storage, EP showed the lowest means of EM and FS ($P<.05$). Among RC materials, CE had the lowest mean for EM and the highest for FS, while EN presented the highest EM mean and the lowest for FS, for both storage periods ($P<.05$). In general, one-year EM means were higher than 24-h ($P<.05$), except for CE ($P>.05$). After one year, FS reduced significantly compared to 24-h ($P<.05$), except for EN ($P>.05$).

Elemental composition (wt%) of EP using EDS method identified the presence of: C (38.8), O (38.5), Si (20.6), Ca (0.7), Al (0.6), Na (0.4), Ba (0.2), Cl (0.2) and K (<0.1) (Fig. 6A, 6E; Table 5); and SEM micrographs showed particles larger than 10 μm in size (Fig. 7A, 8A). EN contained mainly: O (37.3), C (33.1), Si (15.4), Al (6.7), Na (3.7), K (3.2), In (0.4), Ca (0.1) and Sn (0.1) (Fig. 6B, 6F; Table 5); and a different structural pattern, due to the polymer infiltration in a ceramic network (Fig. 7B, 8B). For LU: O (38.2), C (30.8), Si (19.5), Zr (11.5) and Na (<0.1) (Fig. 6C, 6G; Table 5); while CE composition consisted of: O (32.3), C (32.3), Ba (17.2), Si (15.4), Al (2.7), Ca (0.1) and Cl (<0.1) (Fig. 6D, 6H; Table 5). LU and CE presented particles smaller than 2 μm in size, approximately. Spherical particles were observed for LU (Fig. 7C, 8C) and CE presented the smallest ones compared to other indirect materials (Fig. 7D, 8D).

Discussion

The first null hypothesis was accepted because no significant increase in SBS of resin cements to indirect materials was found with NTAP treatment, regardless of the storage time, type of resin cement and indirect material evaluated. Thus, clinicians must follow manufacturers' instructions in order to obtain optimum adhesion of resin cements with regards to indirect materials tested. NTAP is an artificial plasma created by partial ionized gas, which generates highly reactive particles (ions, electrons, free radicals and electronically excited neutrals) that can increase surface energy and decrease contact angle.[18] In Restorative Dentistry, NTAP has been used to improve adhesion to zirconia[19,20] and dentin[13,21,22]. These studies proved that NTAP treatment has notably enhanced wettability and bond strength of resin cements to zirconia,[23,24] and increased bond strength of adhesives and their penetration into dentin.[25,26] In this study, NTAP was applied to resin-based CAD-CAM materials to improve adhesion; however, the results showed lower bond strength to plasma-treated surfaces compared to manufacturers' instructions. It is well-known that plasmas can be used to initiate polymerization,[27,28] if NTAP application indeed increased the degree of conversion of materials tested, the number of residual monomers free to bond might have decreased, resulting on a less efficient bonding procedure of resin cements to indirect materials. Investigations of NTAP potential of inducing post-polymerization of CAD-CAM composites and hybrid materials, may lead to a better understanding of these results.

With regards to resin cements tested, RelyX Ultimate yielded greater results than Panavia V5 for most experimental groups. Panavia V5 is a conventional dual-cure resin cement that needs an adhesive (Tooth Primer; Kuraray Noritake) to promote bonding to dental substrates. Manufacturer claims that this adhesive contributes to resin cement polymerization, but is indicated for treating the tooth surface only. As our study design did not involve enamel or dentin, Tooth Primer was not used in any experimental group, which may have affected the quality of cement polymerization; and consequently, its mechanical properties and bonding process. Likewise, the lower content of fillers, specially zirconia and alumina for Panavia V5 compared to RelyX Ultimate might have some influence on its mechanical behavior.[29]

This study brings new information about the use of two resin cements applied to resin-based CAD-CAM materials, which have never been reported in Dental Literature. Many studies have evaluated the bond strength of resin cements to CAD-CAM composites and hybrid materials;[16,30–35] however, few have analyzed the effect of artificial ageing.[16,30,35] In these studies, water storage and thermal cycling resulted in significant decrease in bond strength of resin cements to RCs,[16,30,35] which is in accordance to most of the results of this study. Due to water immersion, methacrylates and filler-polymer interface can be hydrolyzed,[7] which contributes to the degradation of the polymer chain. Besides, water sorption allows free monomers and inactive polymerization promoters to be eluted,[36] which reduces both adhesive and resin cement mechanical properties, making them more susceptible to fail.

The conventional composite (EP) was selected as a control group, because it is not obtained through CAD-CAM technology. This material displayed 100% of cohesive failures of its structure when used according to manufacturer's instructions, regardless of the storage period and resin cement. This behavior was not present on CAD-CAM materials tested, except for RelyX Ultimate applied to CE. The greater number of free monomers on the surface of EP, due to polymerization activated by light only, may have resulted on a strong adhesion of resin cements to its surface,[37] which forced the failure to occur within the composite structure. Also, the cohesive strength of this material may be lower than CAD-CAM materials, as observed in mechanical properties evaluated in this study. The industrial polymerization of CAD-CAM blocks following ideal conditions of temperature and pressure, leads to a higher degree of conversion of the organic matrix and better mechanical properties. In addition, shear bond strength test is known to induced cohesive failures within bonded material due to concentration of stress on the base of the specimen,[38] as observed in some samples after testing.

In general, NTAP and NTAP + BA groups had a greater number of adhesive failures, while MI groups presented mixed and cohesive failures more often. This corroborates with high bond strength means for MI groups, in which RelyX Ultimate bonded to CE for both storage periods displayed 100% of cohesive failure, while Panavia V5 had more than 85% of adhesive failures. These differences can also be noticed for bond strength results of RelyX Ultimate that were approximately the double compared to Panavia V5.

The second null hypothesis was rejected since CAD-CAM materials presented higher FS and EM than those obtained for EP. This conventional indirect composite showed the lowest EM (3.4 GPa) and FS (72.1 MPa) at 24 h. This may be related to the lower degree of conversion of EP compared to industrial-polymerized CAD-CAM blocks, which makes the conventional indirect composite more resilient and flexible.[39] Among CAD-CAM materials, EN displayed the highest EM (23.3 GPa), which is close to that of dentin (17.7 - 29.8 GPa).[40] The porous interconnected feldspar network infiltrated with organic polymeric chain results in a rigid material, with low flexibility and resilience. As revealed by SEM images (Fig. 7C, 7D, 8C, 8D) LU and CE have their inorganic particles disperse in the organic matrix, which can result on a better tension distribution along their structures leading to a higher flexibility, thus a lower EM (10.4 and 7.0 GPa, respectively). EM of LU was higher than CE, which may be related to its higher filler concentration[41] (80 wt%) compared to CE (71 wt%) (Table 1).

At one-year storage period the FS of EN and LU had no significant difference, while EP presented the lowest FS mean, probably due to its polymerization mode.[42] Among CAD-CAM blocks, CE always showed higher FS than EN and LU. However, it has been proved that LU presents higher fracture toughness than EN and CE.[43] In addition, EN and LU contain higher inorganic content when compared to CE (Table 1), which might be expect to result on greater flexural strength.[41] This finding may be attributed to the microstructure of these materials (Fig. 7, 8). CE presents uniform and small fillers highly prone to a homogeneous tension distribution, while LU exhibits varied size range of fillers. On the other hand, EN is a hybrid material with an organic matrix of UDMA and TEGDMA injected into a feldspar ceramic network, which tend to have a none homogeneous tension distribution. The difference on these materials behavior is clearly observed on the stress-strain plot displayed in Figure 5. EM and FS results from this study are similar to those obtained by other studies, despite methodological differences, especially concerning specimen dimensions.[3,43–45]

Very few studies evaluated the effect of aging on EM and FS of CAD-CAM composites and hybrid materials.[44,45] Moreover, none of them evaluated water storage for periods longer than 7 days.[44] This is the first study to evaluate the effect of one-year water-storage on EM and FS of these materials. The findings revealed

an increase on EM and a decrease on FS, except for EM of CE and FS of EN. Studies that evaluated the same materials after 10,000 thermal cycles obtained similar results for FS, but different results for EM as they decreased for LU and CE, while no statistical difference was found for EN.[44,45] The volumetric variation induced by temperature shift during thermal cycling may induce some damage on the polymeric chain, breaking it in smaller oligomers and reducing cross-linking, which increase the material resilience, consequently reducing EM.[46] On the other hand, one-year water storage performed in our study can induce water sorption and monomer leaching that can plasticize the polymeric materials and increase their EM.[7]

EP was the most affected material with a 17.6% increase on EM and 37.6% decrease on FS. It is known that degree of conversion is inversely proportional to water sorption.[47,48] The lower degree of conversion of EP might have led to a greater water sorption, resulting on a higher deterioration of its structure. EN had the highest increase on EM among CAD-CAM materials (21.5%), while LU suffered the greatest decrease on FS (21.5%). It may be hypothesized that water absorbed by LU organic matrix can hydrolyze the coupling agent molecule of zirconium silicate filler, which is not effectively silanized due to its high inorganic content.[49] CE seemed to be the least affected material, as the EM suffered no significant increase, and FS decreased only 13%. Thus, according to the literature, the changes in mechanical properties of indirect materials tested by one-year water storage induces hydrolytic degradation of polymers, which reduces its resistance, at the same time that lixiviates residual monomers from its structure, increasing its stiffness.[7] However, the adhesion and mechanical behaviors of each material depends on their compositions and microstructures that varied among them.

Besides carbon and oxygen, silicon was the unique chemical element identified in all materials. Only EN has indication of indium and strontium on its composition, which was not detected by previous studies.[1,44,50] In these studies zirconium[1,50] and yttrium[44] have been identified at EN structure. EP and CE presented traditional types of glasses, such as, aluminum and barium, but contained different filler particle sizes. Only LU contained zirconium, which is related to strength of the material, but interferes negatively on bond strength of resin cement. Nevertheless, although materials showed different compositions and microstructures, they did not seem to influence bond strength of resin cements.

This study evaluated only the effect of hydrolytic degradation in resin-based CAD-CAM materials and some clinical conditions were not reproduced, but have a critical influence on clinical performance of these materials. Occlusal loading, saliva, pH, temperature variation and biofilm formation are the main clinical conditions that can validate the clinical use of different restorative materials based on in vitro studies. Although the direct correlation with clinical performance of this type of indirect restoration is not possible, the decrease on their bond and flexural strength and increase on elastic modulus in the presence of water found in our study, suggest a tendency of failure due to debonding and fracture overtime.

Conclusions

Based on the findings of this in vitro study, the following conclusions were drawn:

1. Surface treatment of resin-based CAD-CAM materials before cementation with resin cements should follow manufacturers' instructions, with no significant benefit on the use of NTAP associated or not with a bonding agent.
2. In general, RelyX Ultimate obtained greater SBS than Panavia V5, for both storage periods.
3. One-year water storage decreased SBS of resin cements to indirect materials for most experimental groups.
4. CAD-CAM blocks showed higher EM and FS compared to conventional indirect composite and among CAD-CAM blocks, CE presented the lowest EM and highest FS, while EN showed the highest EM and lowest FS, for all storage periods.
5. Water storage for one-year tended to increase EM and decrease FS.
6. Different compositions and microstructures were detected among the resin-based indirect materials.

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Table 1. Materials, manufacturers, compositions and batch numbers used in this study.

Material	Manufacturer	Composition	Batch #
Epricord	Kuraray Noritake	24 wt% organic - TEGDMA, UDMA | 76 wt% inorganic: pre-polymerized organic filler, glass and colloidal silica filler. Dl-camphorquinone, initiators, accelerators, pigments	00149A 00445A
Enamic	Vita Zahnfabrik	14 wt% organic - UDMA e TEGDMA | 86 wt% inorganic: feldspar ceramic and alumina	45010 45360 43130
Lava Ultimate	3M ESPE	20 wt% organic: Bis-GMA, UDMA, Bis-EMA, TEGDMA | 80 wt% inorganic - silica, zirconia nanoparticules	N804141
Cerasmart	GC Dental Products	29 wt% organic: Bis-MEPP, UDMA, dimethacrilate | 71 wt% inorganic: silica, barium nanoparticules	1504281
Panavia V5	Kuraray Noritake	*Paste A*: Bis-GMA, TEGDMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, initiators, accelerators, silanated barium glass filler, silanated, fluoroaluminosilicat glass filler, colloidal silica *Paste B*: Bis-GMA, hydrophobic aromatic dimethacrylate, hydrophilic aliphatic dimethacrylate, silanated barium glass filler, silanated aluminum oxide filler, accelerators, dl-camphorquinone, pigments	5J0032 6A0003 1U0001
RelyX Ultimate	3M ESPE	*Base paste*: TEGDMA, Silane-treated glass powder, 2-propenoic acid, 2-methyl-, reaction products with 2-hydroxy-1,3-propanedyl dimethacrylate and phosphorus oxide, silane-treated silica, oxide glass chemicals, sodium persulfate, tertbutyl peroxy-3,5,5- trimethylhexanoate, copper acetate monohydrate *Catalyst paste*: Substituted dimethacrylate, 1,12-dodecane dimethacrylate, Silane-treated glass powder, silane-treated silica, 1-benzyl-5-phentyl-barbic-acid, calcium salt, sodium p-toluenesulfinate, 2-propenic acid, 2-methyl-, di-2,1-ethanediyl ester, calcium hydroxide, titanium dioxide	621762
Condac Porcelana 5%	FGM	5% hydrofluoric acid	50815
Clearfill Ceramic Primer Plus	Kuraray Noritake	MDP, ethanol, 3-trimethoxysilylpropyl methacrylate	4R0015 5N0003
RelyX Ceramic Primer	3M ESPE	Ethanol, water, methacryloxypropyltrimethoxysilane	N555194 N822741
Ceramic Primer II	GC Dental Products	MDP, ethanol, 3-trimethoxysilylpropyl methacrylate, 2,2' -ethylenedioxydiethyl dimethacrylate.	1507162
Scotchbond Universal	3M ESPE	HEMA, Bis-GMA, MDP, decamethylene dimethacrylate, silane treated silica, copolymer of propenoic and itaconic acid, (dimethylamino)ethyl methacrylate, camphorquinone, dimethylaminobenzoat (-4), methyl ethyl ketone, ethanol, water	610586
Scotchbond Multi-Purpose	3M ESPE	*Adhesive (#3)*: Bis-GMA, HEMA, triphenylantimony	N733996 N515442

TEGDMA, triethylene glycol dimethacrylate; UDMA, urethane dimethacrylate; Bis-GMA, bisphenol a diglycidyl ether dimethacrylate; Bis-EMA, ethoxylated bisphenol A glycol dimethacrylate; Bis-MEPP, bis[2-(methacryloyloxy)ethyl] phosphate; MDP, 10-methacryloxydecyl dihydrogen phosphate; HEMA, hydroxyethyl methacrylate.

Table 2. Experimental groups' distribution and surface treatment descriptions (n = 10).

Indirect Material	Treatment	Resin Cement	
		Panavia V5	**RelyX Ultimate**
Epricord	Manufacturers' Instructions	Sandblasting + Clearfill Ceramic Primer	Sandblasting + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose
	Plasma	Sandblasting + Plasma	Sandblasting + Plasma
	Plasma + Bonding agent	Plasma + Clearfill Ceramic Primer	Plasma + Scotchbond Universal
Enamic	Manufacturers' Instructions	Hydrofluoric acid (HF) 5% 60s + Clearfill Ceramic Primer	HF 5% 60s + RelyX Ceramic Primer + Adhesive-Scotchbond Multipurpose
	Plasma	Sandblasting + Plasma	Sandblasting + Plasma
	Plasma + Bonding agent	Plasma + Clearfill Ceramic Primer	Plasma + Scotchbond Universal
Lava Ultimate	Manufacturers' Instructions	Sandblasting + Clearfill Ceramic Primer	Sandblasting + Scotchbond Universal
	Plasma	Sandblasting + Plasma	Sandblasting + Plasma
	Plasma + Bonding agent	Plasma + Clearfill Ceramic Primer	Plasma + Scotchbond Universal
Cerasmart	Manufacturers' Instructions	Sandblasting + Ceramic Primer II	Sandblasting + Ceramic Primer II
	Plasma	Sandblasting + Plasma	Sandblasting + Plasma
	Plasma + Bonding agent	Plasma + Ceramic Primer II	Plasma + Ceramic Primer II

Table 3. SBS means (SD) of resin cements bonded to the indirect resin materials according to each experimental group (in MPa).

Resin Cement	Indirect Material	Storage Period					
		24 h			1 Year		
		Treatment			Treatment		
		Manufacturers' Instructions	Plasma	Plasma + Bonding agent	Manufacturers' Instructions	Plasma	Plasma + Bonding agent
Panavia V5	Epricord	11.6 (2.1) Aa	5.8 (1.5) Ba	2.7 (0.7) Cb	9.0 (1.6) Aa	2.6 (1.4) Ba	1.6 (1.0) Cb
	Enamic	10.0 (2.5) Aa	4.5 (0.9) Bab	5.8 (1.4) Ba	§ 9.1 (1.5) Aa	0.9 (0.8) Cb	2.9 (0.8) Ba
	Lava Ultimate	6.5 (1.3) Ab	4.6 (1.2) Bab	3.1 (0.8) Cb	3.1 (1.5) Ac	0.8 (0.5) Bbc	1.2 (0.6) Bb
	Cerasmart	8.1 (1.4) Ab	3.4 (0.6) Bb	3.6 (1.0) Bb	6.0 (1.8) Ab	0.2 (0.2) Cc	1.3 (0.7) Bb
RelyX Ultimate	Epricord	* 12.6 (2.2) Ac	8.9 (2.1) Bb	10.1 (2.6) Bb	§ 11.5 (1.7) Aab	* 3.1 (2.0) Bc	* 2.3 (0.8) Bb
	Enamic	15.7 (4.1) Aa	12.7 (3.2) Ba	10.1 (2.1) Cb	11.8 (2.7) Aab	6.9 (2.3) Ba	7.5 (1.7) Ba
	Lava Ultimate	13.0 (3.1) Abc	10.1 (2.2) Bb	9.8 (2.0) Bb	10.9 (2.8) Ab	7.1 (0.8) Ba	6.9 (2.5) Ba
	Cerasmart	14.9 (2.6) Aab	9.5 (2.3) Cb	12.7 (3.3) Ba	§ 13.1 (2.1) Aa	4.2 (1.2) Cb	8.6 (2.8) Ba

*Do not differ from Panavia V5 within the same treatment, indirect resin and storage period (P<.05). § Do not differ from 24h within the same resin cement, indirect resin and treatment (P<.05). Means followed by different letters (uppercase letters compare treatments within the same storage period, indirect resin and resin cement and lowercase letters compare resins within the same storage period, treatment and resin cement) differ among them (by Tukey's test. P<.05).

Table 4. Elastic modulus (GPa) and flexural strength (MPa) means (SD) for resin materials tested after 24 hours and one-year water-storage.

Indirect Material	Elastic Modulus		Flexural Strength	
	24 h	**1 Year**	**24 h**	**1 Year**
Epricord	3.4 (0.3) Bd	4.0 (0.4) Ad	72.1 (10.7) Ad	45.0 (7.6) Bc
Enamic	23.3 (1.7) Ba	28.3 (2.1) Aa	133.3 (16.0) Ac	118.9 (9.0) Ab
Lava Ultimate	10.4 (0.6) Bb	12.0 (0.9) Ab	164.8 (12.8) Ab	129.4 (15.4) Bb
Cerasmart	7.0 (0.7) Ac	7.8 (0.9) Ac	197.0 (19.1) Aa	171.4 (15.4) Ba

Means followed by different uppercase (horizontal - comparing storage periods for the same material) and lowercase (vertical - compare materials for the same storage period) letters are statistically different (P<.05).

Table 5. Chemical elemental concentrations (SD) from EDS analyses in weight percentage.

Indirect Material	Element analysis (wt%)											
	C	**O**	**Si**	**Al**	**Na**	**Ca**	**Ba**	**K**	**Cl**	**Zr**	**In**	**Sn**
Epricord	38.8 (4.9)	38.5 (3.1)	20.6 (2.1)	0.6 (0.8)	0.4 (0.2)	0.7 (0.8)	0.2 (0.4)	<0.1 (0.1)	0.2 (0.3)	-	-	-
Enamic	33.1 (2.7)	37.3 (1.8)	15.4 (0.8)	6.7 (0.3)	3.7 (0.3)	0.1 (0.2)	-	3.2 (0.5)	-	-	0.4 (0.2)	0.1 (0.2)
Lava Ultimate	30.8 (0.4)	38.2 (1.3)	19.5 (0.9)	-	<0.1 (0.1)	-	-	-	-	11.5 (0.6)	-	-
Cerasmart	32.3 (1.3)	32.3 (5.4)	15.4 (0.9)	2.7 (0.1)	-	0.1 (0.1)	17.2 (4.2)	-	<0.1 (0.0)	-	-	-

Figures

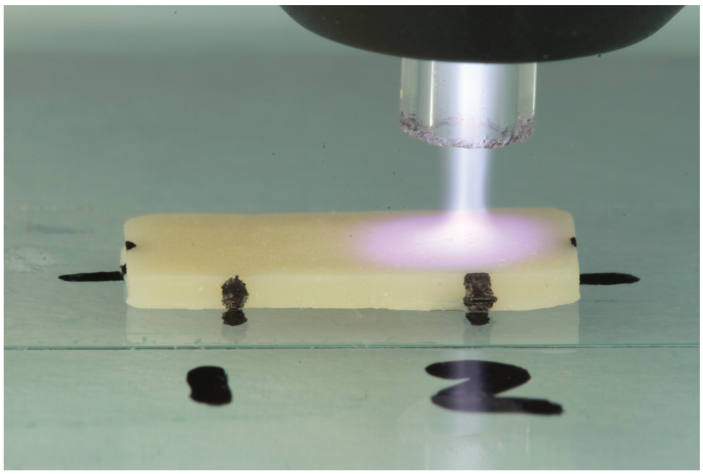

Figure 1. NTAP application on indirect material.

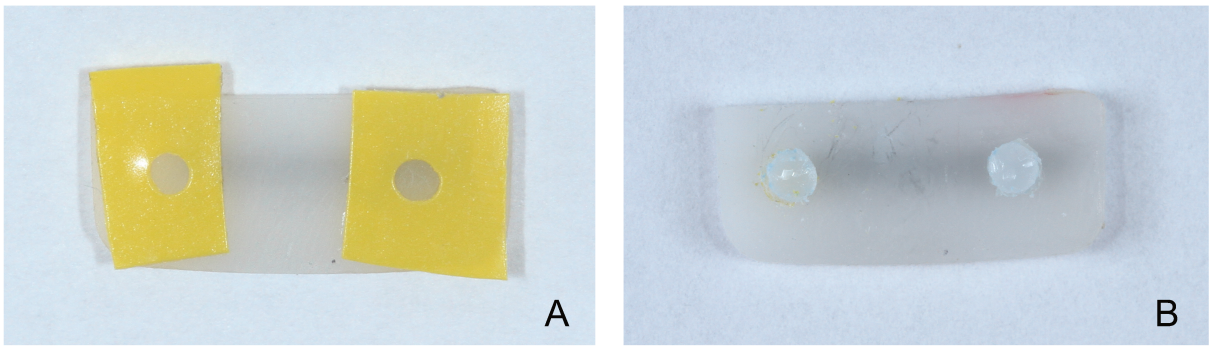

Figure 2. A, Delimitation of bonding area with tape. B, Resin cylinders after bonding procedure.

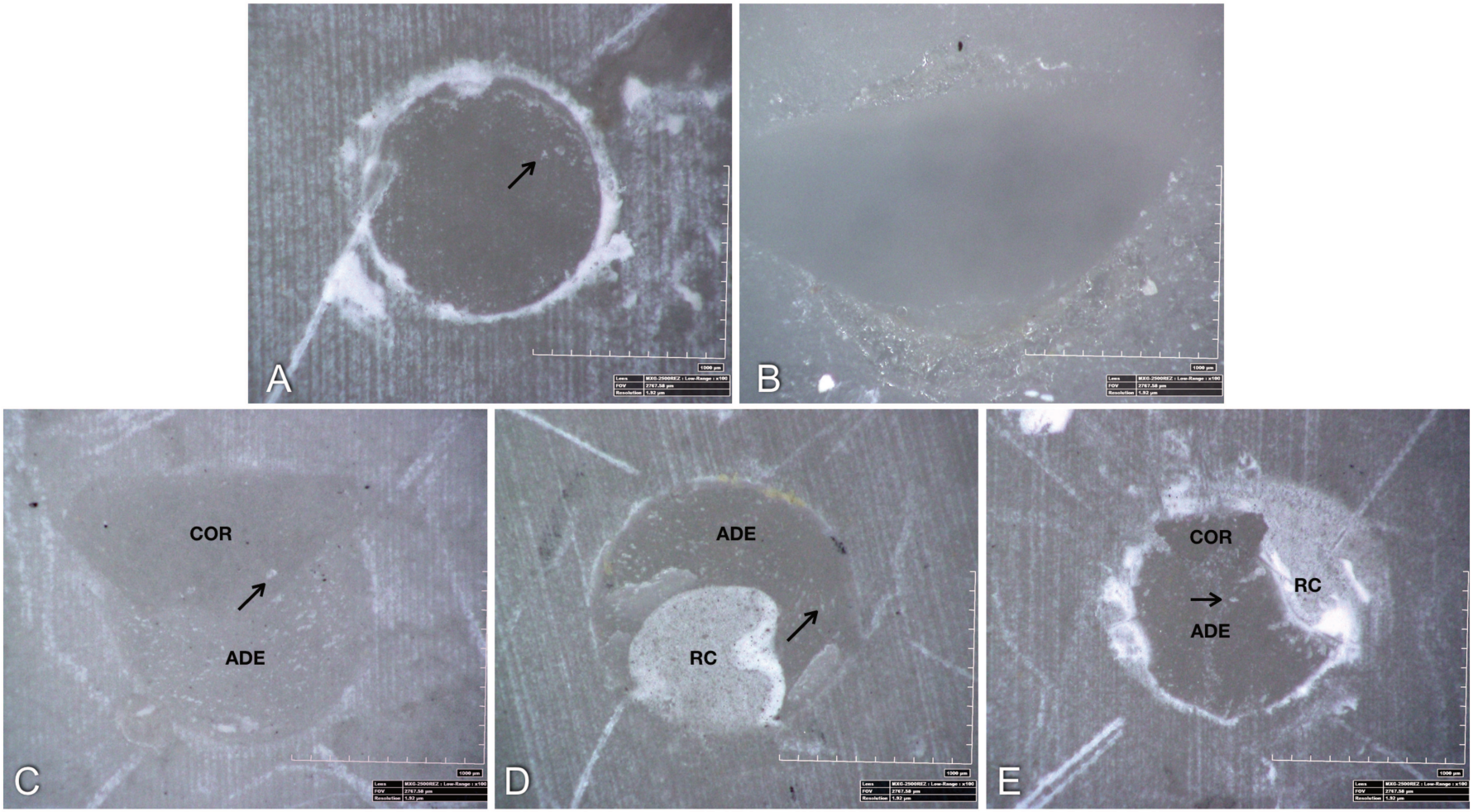

Figure 3. Representative micrographs at x100 magnification of each failure mode: A, Adhesive failure; B, Cohesive failure within indirect resin; C, Mixed failure involving adhesive layer and indirect resin; D, Mixed failure involving adhesive layer and resin cement; E, Mixed failure involving adhesive layer, indirect resin and resin cement. Black arrows indicate remnant of resin cement on the surface of indirect material. COR, cohesive failure within indirect resin; RC, resin cement.

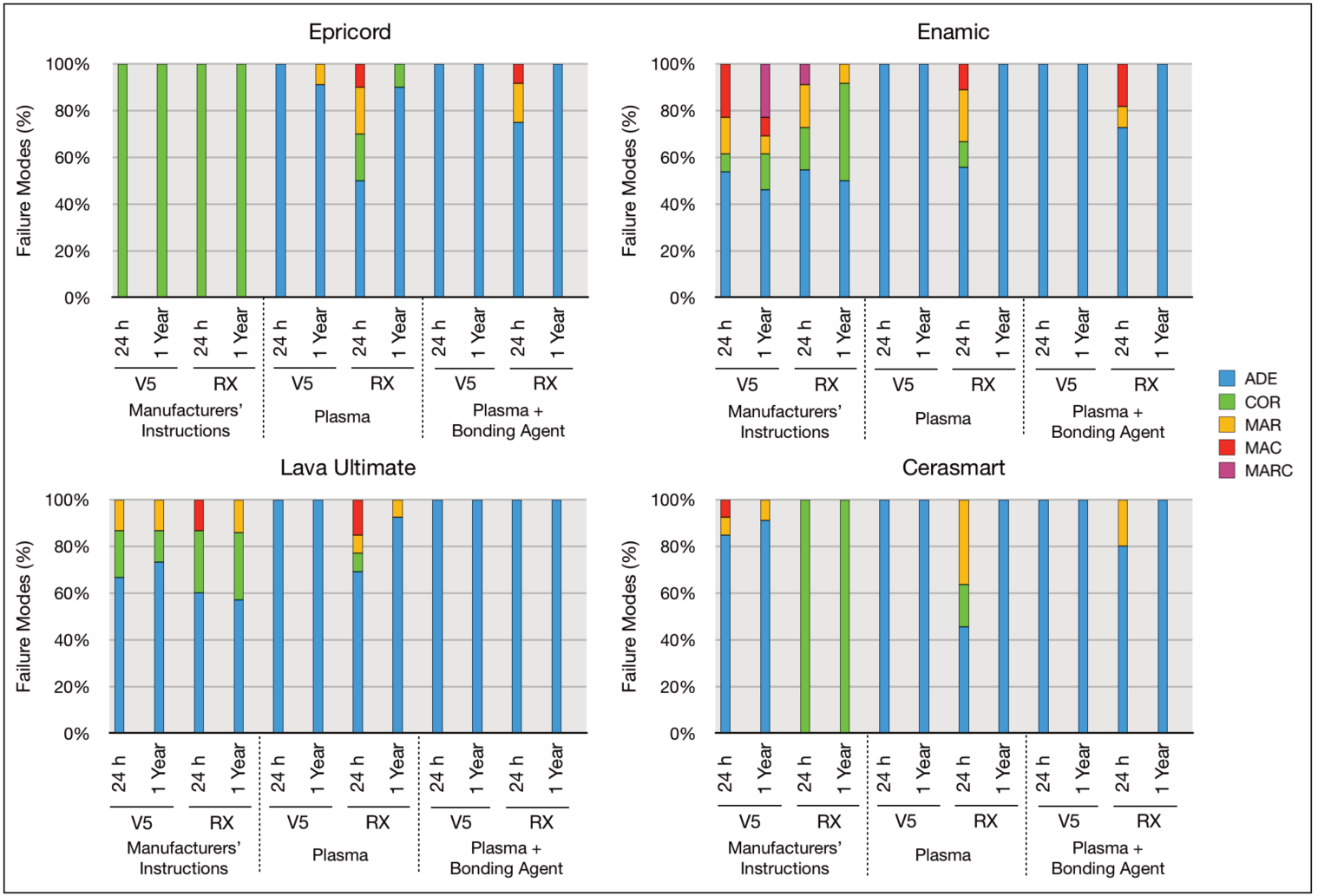

Figure 4. Bar graph presentation of proportional prevalence of fracture modes for resin cements bonded to indirect materials. V5, Panavia V5; RX, RelyX Ultimate; ADE, Adhesive failure; COR, Cohesive failure within indirect resin; MAR, Mixed failure involving adhesive layer and indirect resin; MAC, Mixed failure involving adhesive layer and resin cement; MARC, Mixed failure involving adhesive layer, indirect resin and resin cement.

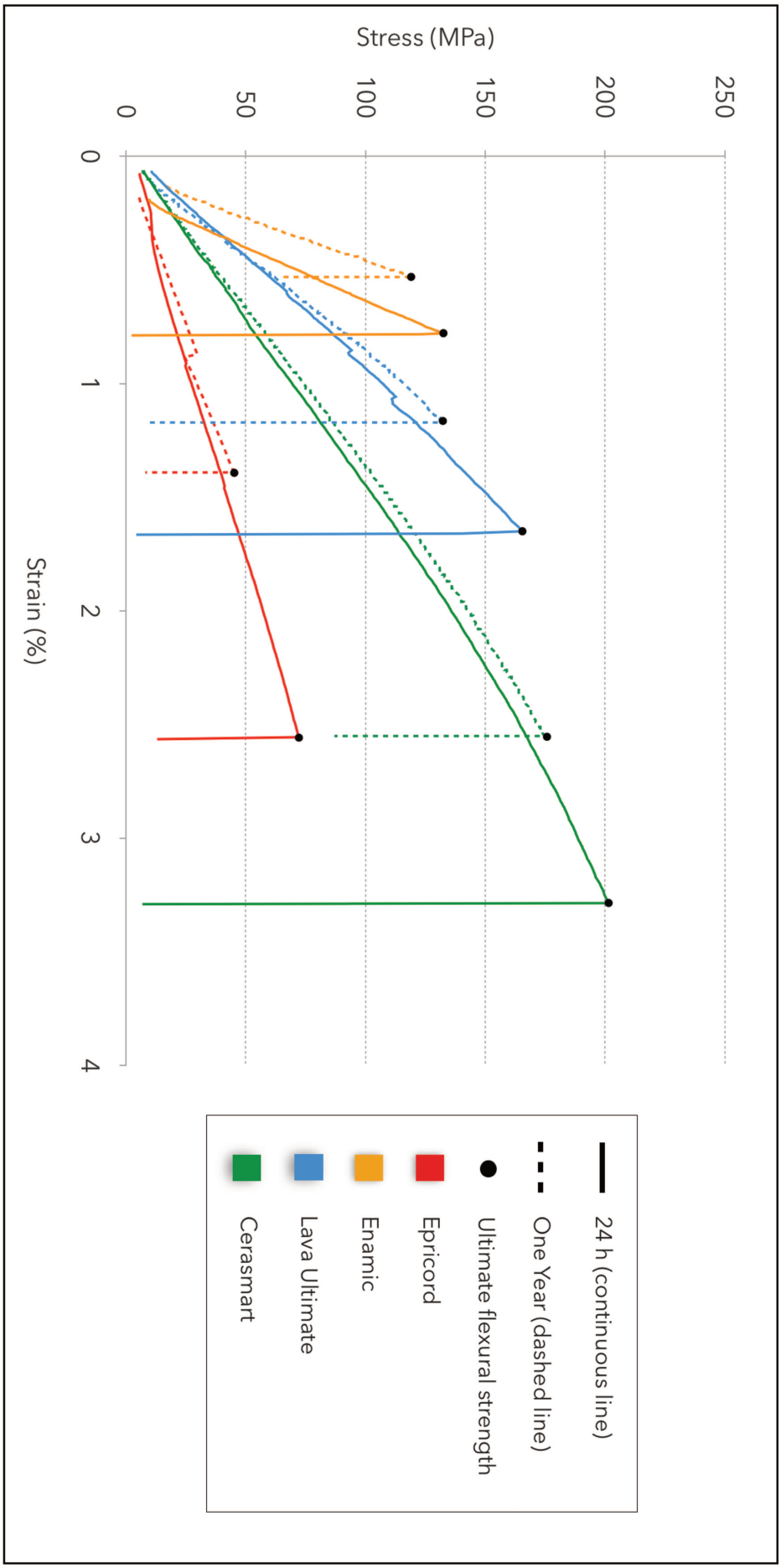

Figure 5. Stress-strain plot of indirect materials submitted to three-point flexural test after 24 h and one-year water storage.

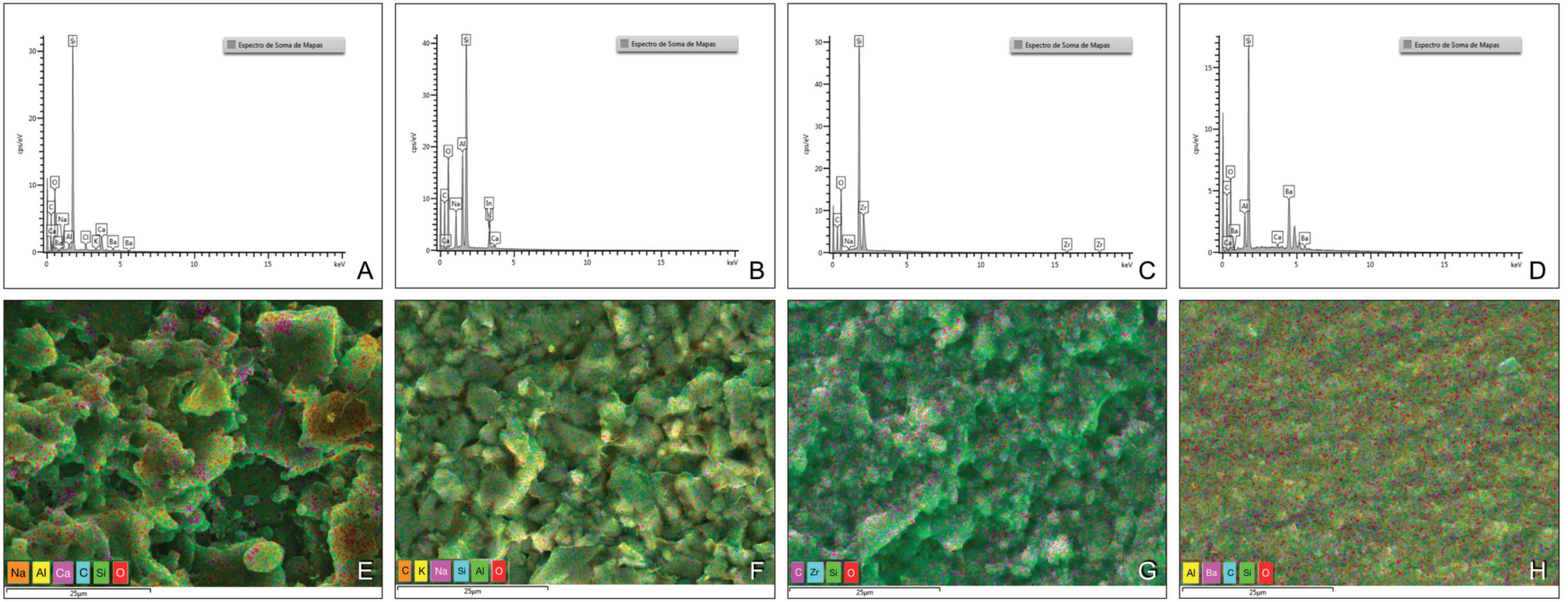

Figure 6. Identified elements by EDS analyses (A-D) and EDS mapping at X2000 magnification (E-H) of indirect materials: Epricord (A,E), Enamic (B,F), Lava Ultimate (C,G) and Cerasmart (D,H).

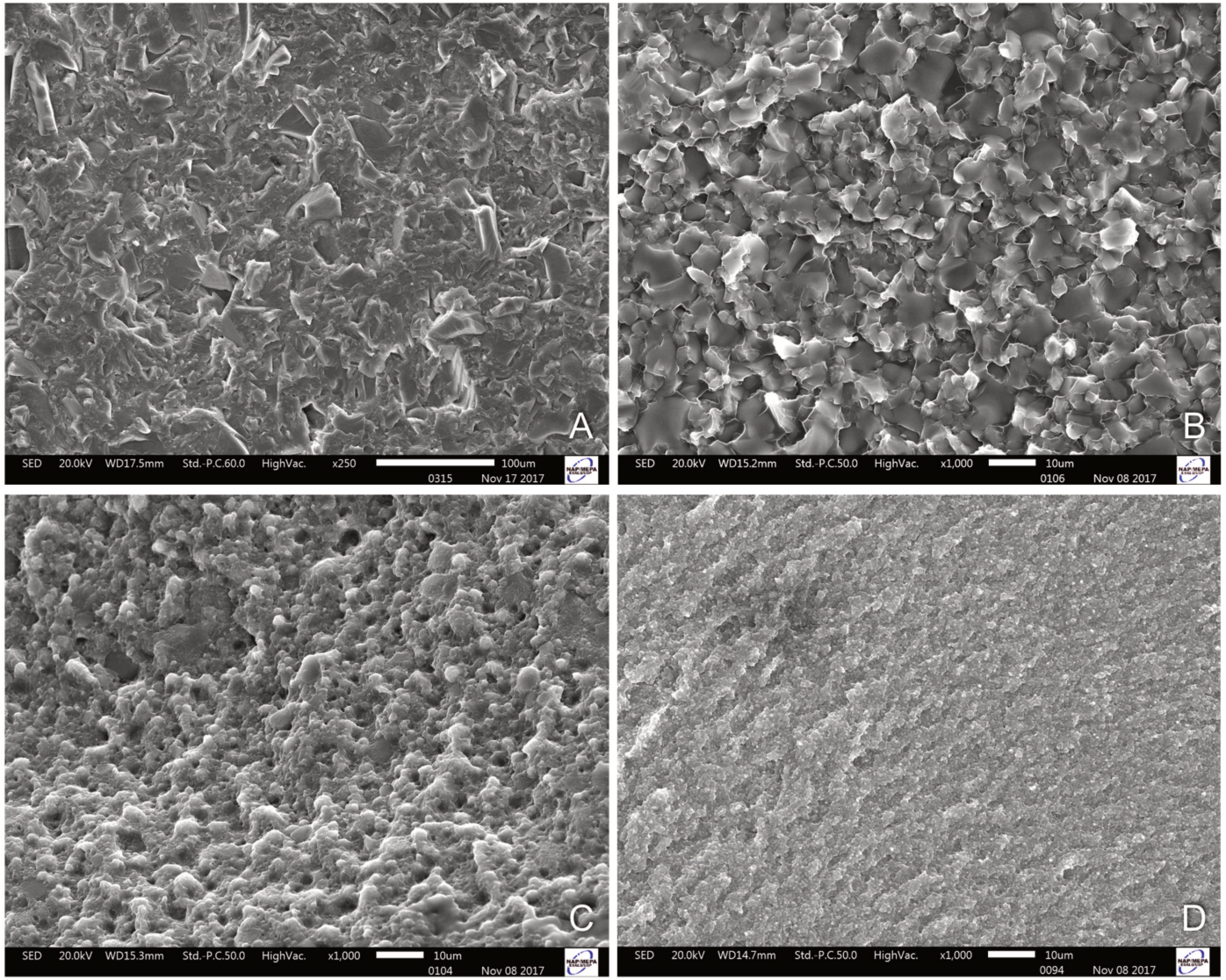

Figure 7. Representative SEM micrographs of fractured surfaces of indirect materials at x250 (A, Epricord) and x1000 (B, Enamic; C, Lava Ultimate; D, Cerasmart).

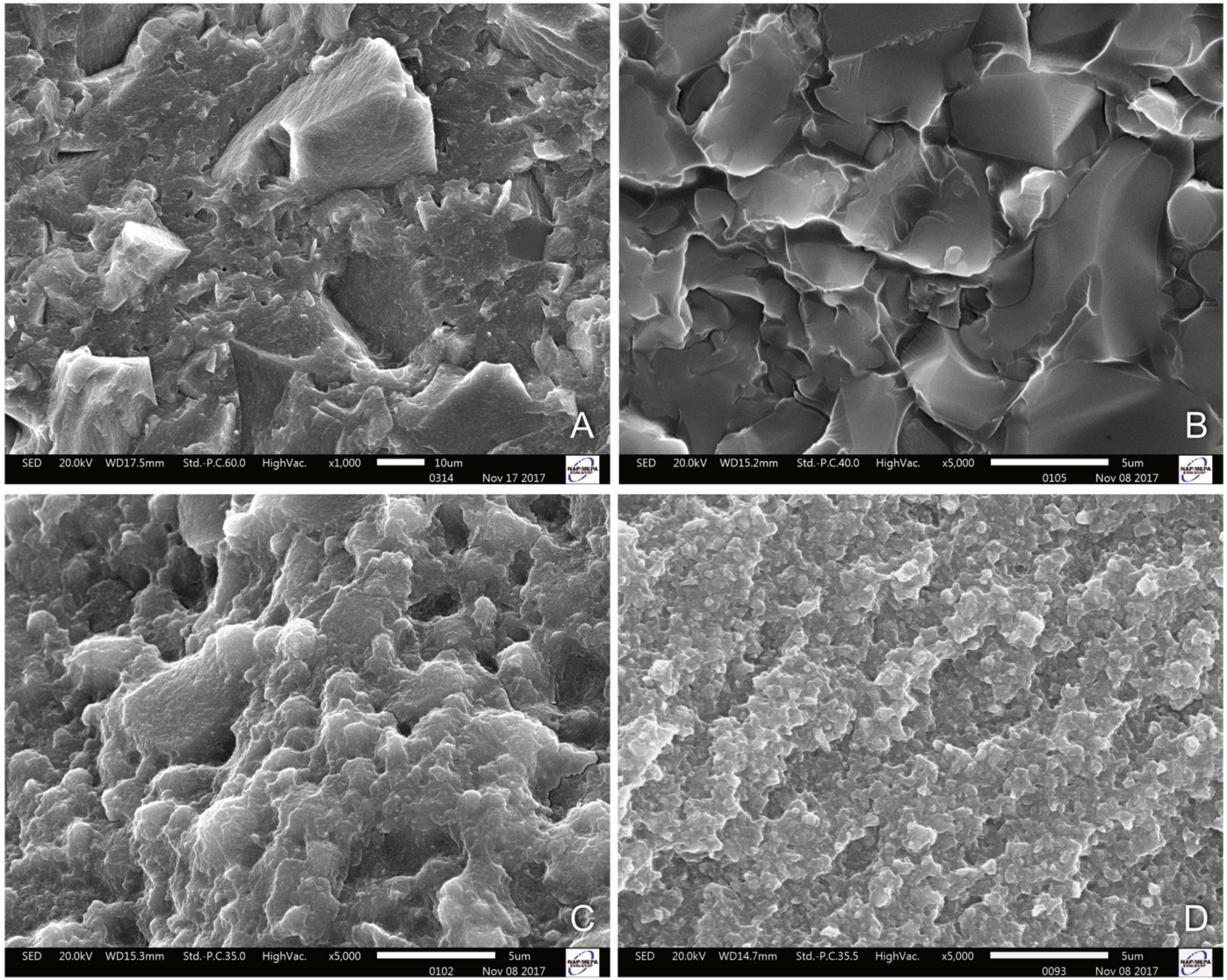

Figure 8. Representative SEM micrographs of fractured surface of indirect materials at x1000 (A, Epricord) and x5000 (B, Enamic; C, Lava Ultimate; D, Cerasmart).

CONCLUSÃO

Baseado nos resultados deste estudo *in vitro*, conclui-se que:

1. O tratamento de superfície dos materiais CAD/CAM à base de resina para cimentação adesiva deve seguir as instruções dos fabricantes, sem benefício aparente no uso do NTAP associado ou não com um agente de união.
2. Em geral, o RelyX Ultimate obteve maior resistência de união que o Panavia V5, em ambos tempos de avaliação.
3. Um ano de armazenamento em água reduziu a resistência de união por cisalhamento dos cimentos resinosos aos materiais indiretos, na maioria dos grupos experimentais.
4. Os materiais CAD/CAM obtiveram maior módulo de elasticidade e resistência flexural que o compósito convencional indireto. Dentre os materiais CAD/CAM, Cerasmart apresentou o menor módulo de elasticidade e a maior resistência flexural, enquanto Enamic obteve maior módulo de elasticidade e menos resistência flexural, para ambos períodos de armazenamento em água.
5. O armazenamento em água por um ano tende a aumentar o módulo de elasticidade e diminuir a resistência flexural.
6. Os materiais indiretos apresentaram diferenças composicionais e estruturais.

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