UNESP - UNIVERSIDADE ESTADUAL PAULISTA

"JÚLIO DE MESQUITA FILHO"

FACULDADE DE ODONTOLOGIA DE ARARAQUARA

FILIPE DE OLIVEIRA ABI RACHED

CARACTERIZAÇÃO DE SUPERFÍCIE, ADESIVA E MECÂNICA DE UMA ZIRCÔNIA PARCIALMENTE ESTABILIZADA POR ÍTRIA EM FUNÇÃO DA PARTÍCULA OU DO MOMENTO DO JATEAMENTO

ARARAQUARA

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Orientadora: Profa. Dra. Renata Garcia Fonseca

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Filipe de Oliveira Abi Rached

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Araraquara, 20 de março de 2014.

DADOS CURRICULARES

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Aos meus avós

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"Sonho que se sonha só...É só um sonho que se sonha só

Mas sonho que se sonha junto é realidade"

Raul Seixas

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"Um irmão pode não ser um amigo, mas um amigo será sempre um irmão."

Benjamin Franklin

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“A alegria está na luta, na tentativa, no sofrimento envolvido. Não na vitória propriamente dita.”

Mahatma Gandhi (1869-1948)

Resumo

Abi Rached FO. Caracterização de superfície, adesiva e mecânica de uma zircônia parcialmente estabilizada por ítria em função da partícula ou do momento do jateamento [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2014.

RESUMO

A retenção mecânica e a longevidade das restaurações indiretas com infraestrutura de zircônia podem ser favorecidas pela cimentação adesiva. No entanto, como a zircônia é uma cerâmica cristalina, tratamentos de superfície alternativos aos convencionais (ácido fluorídrico e silanização) devem ser utilizados com o objetivo de estabelecer união mecânica e química na interface cimento resinoso/restauração. Dentre os tratamentos promotores de retenção micromecânica, o jateamento é ainda um dos métodos mais eficazes e fáceis. Esse procedimento pode ser realizado com partículas de óxido de alumínio/alumina (Al_2O_3) convencionais e/ou revestidas por sílica de diferentes tamanhos e, portanto, diferentes comportamentos quanto à rugosidade e à área de superfície disponível para adesão, bem como ao molhamento por agentes de união, podem ser observados. Apesar do seu papel benéfico na resistência de união da interface cimento resinoso/zircônia e até mesmo na resistência mecânica imediata da zircônia, sabe-se que o jateamento favorece a criação de microtrincas superficiais, as quais, em longo prazo, sob as condições adversas da cavidade oral, tendem a se propagar pelo corpo do material, reduzindo sua resistência mecânica ou até mesmo promovendo sua falha catastrófica. Considerando que o protocolo convencional preconiza o jateamento da zircônia

sinterizada, uma alternativa seria realizá-lo na zircônia previamente à sua sinterização, uma vez que ela seria mais propícia à criação de retenções micromecânicas e, possivelmente, os efeitos da severidade do jateamento e seus danos na resistência mecânica do material, em longo prazo, seriam minimizados. Levando-se em consideração os aspectos ressaltados, este estudo teve como objetivos: 1) Avaliar o efeito da partícula empregada no jateamento na rugosidade de superfície, molhamento e padrão morfológico de uma zircônia parcialmente estabilizada por ítria, bem como verificar a existência de correlação entre as variáveis dependentes rugosidade e molhamento; 2) Avaliar o efeito do momento do jateamento na caracterização de superfície (rugosidade, morfologia e transformação de fase) e resistência à flexão de uma zircônia parcialmente estabilizada por ítria, sua resistência de união ao cisalhamento com um cimento resinoso e o modo de fratura predominante na área adesiva. Também verificou-se a existência de correlação entre as variáveis dependentes rugosidade e resistência de união. No primeiro estudo, espécimes (14 × 14 × 1,4 mm) da zircônia Lava (3M ESPE AG) foram obtidos e, com exceção do grupo controle (n=20), jateados com uma das seguintes partículas (n=20): 1) Al_2O_3 de 50 μm; 2) Al_2O_3 de 120 μm; 3) Al_2O_3 de 250 μm; 4) Al_2O_3 de 30 μm revestida por sílica (Rocatec Soft); 5) Al_2O_3 de 110 μm revestida por sílica (Rocatec Plus); 6) Al_2O_3 de 120 μm e Rocatec Plus. As análises de rugosidade (Ra) e molhamento (pelo silano RelyX Ceramic Primer) foram realizadas nos mesmos espécimes. Para a análise da morfologia de superfície por microscopia eletrônica de varredura (MEV), foram preparados dois espécimes adicionais (6,0 × 6,0 × 1,0 mm) para cada grupo. Os dados de Ra (μm) foram analisados por ANOVA a 1 critério e teste Dunnett C (α=0,05), e os dados de molhamento por ANOVA a 1 critério

(α=0,05). O teste de correlação de Spearman foi aplicado para verificar uma possível correlação entre rugosidade e molhamento. Com exceção dos grupos jateados com Al_2O_3 de 120 μm, os demais apresentaram valores de rugosidade estatisticamente diferentes. Com relação ao molhamento, não houve diferença estatística entre os grupos. Não foi observada correlação (r_s=-0,09, *P*=0,27) entre rugosidade e molhamento. A partícula influenciou a morfologia de superfície da zircônia. O aumento da rugosidade pareceu acompanhar o aumento do tamanho das partículas. A análise de MEV mostrou que diferentes partículas promoveram diferenças nos padrões morfológicos. No segundo estudo, espécimes da zircônia Lava foram jateados com Al_2O_3 de 50 μm depois (DS); antes (AS); ou antes e depois (ADS) da sinterização da zircônia. Para a rugosidade (Ra), trinta espécimes (10 × 10 × 3,0 mm) (n=10) foram analisados em rugosímetro. Discos de resina composta (n=30) foram cimentados com RelyX ARC (3M ESPE) sobre estes espécimes e armazenados em água destilada a 37 ºC por 24 horas previamente ao ensaio de resistência de união ao cisalhamento (RC). O modo de fratura foi avaliado em estereomicroscópio (×20). A morfologia de superfície (n=2) foi avaliada por MEV (×500). Para o ensaio de resistência à flexão (RF) 4 pontos (EMIC DL2000), trinta e nove espécimes em formato de barra (20 × 4,0 × 1,2 mm) (n=13) foram jateados de acordo com os três momentos propostos, e um grupo adicional (não jateado) foi avaliado (n=13). A transformação de fase (n=1) foi analisada pelo refinamento de Rietveld com os dados da difração de raios X. Os dados de Ra (μm) e RC (MPa) foram analisados por ANOVA a 1 critério e teste de Tukey (α=0,05). O teste de correlação de Pearson determinou se havia correlação entre rugosidade e RC. Para os dados de RF (MPa), foram utilizados ANOVA a 1 critério e teste Dunnett

C (α=0,05). O momento do jateamento foi significante (*P*<0,001) para os dados de Ra, RC e RF. Os grupos AS e DS apresentaram a maior (1,3 μm) e menor (0,7 μm) Ra. A maior RC (7,0 MPa) foi observada no grupo DAS, seguido pelo grupo DS (5,4 MPa) e, finalmente, pelo grupo AS (2,6 MPa). Todos os grupos apresentaram 100% de falha adesiva. Uma fraca correlação (r=-0,45, *P*<0,05) foi encontrada entre rugosidade e RC. A morfologia de superfície foi influenciada pelo momento do jateamento. Os grupos não jateado (926,8 MPa) e AS (816,3 MPa) apresentaram valores de RF estatisticamente semelhantes, porém valores menores que os grupos DS (1249,1 MPa) e DAS (1181,4 MPa), sem diferença estatisticamente significante entre eles. Os grupos não jateado, DS, AS e DAS mostraram, respectivamente, percentuais em massa de fase monoclínica de 0,0%, 12,2%, 0,0% e 8,6%. A superfície muito rugosa promovida pelo jateamento antes da sinterização da zircônia prejudicou a união com o cimento resinoso. Os padrões morfológicos após os diferentes momentos do jateamento foram condizentes com a rugosidade de superfície. Considerando a RC e a RF imediatas, o grupo DAS exibiu o melhor desempenho. O jateamento, independentemente do momento, promove transformação de fase tetragonal para monoclínica, enquanto a sinterização tende a zerar o conteúdo de fase monoclínica.

Palavras-chave: Cerâmica, propriedades de superfície, resistência ao cisalhamento, resistência de materiais.

Abstract

Abi Rached FO. Surface, adhesive and mechanical characterization of an yttria partially stabilized tetragonal zirconia according to the particle or the moment of air-abrasion [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2014.

ABSTRACT

The mechanical retention and longevity of indirect restorations with zirconia framework can be favored by the adhesive cementation. However, as zirconia is a crystalline ceramic, alternative surface treatments to the conventional ones (hydrofluoric acid and silanization) should be used with the aim of establishing mechanical and chemical bonding at the resin cement/restoration interface. Among the treatments that promote micromechanical retention, the air-abrasion is still one of the most effective and easy-to-use methods. This procedure can be performed with conventional and/or silica-coated aluminum oxide/alumina (Al_2O_3) particles of different sizes and, therefore, different behaviors regards roughness and available surface area for bonding, and also wettability by bonding agents, may be observed. Despite its beneficial role on the bond strength of the resin cement/zirconia interface and even on the short-term mechanical strength of zirconia, it is known that the air-abrasion favors the creation of surface microcracks, which, in the long term, under the adverse conditions of the oral cavity, tend to propagate towards the bulk of the material, decreasing its mechanical strength or even promoting its catastrophic failure. Whereas the conventional protocol recommends the air-abrasion of the sintered zirconia, one alternative would be to perform it on the zirconia before sintering, since it would

be more favorable for the formation of micromechanical retentions and, possibly, the effects of the severity of the air-abrasion and its damage on the mechanical strength of the material, in the long term, would be minimized. Taking into account the highlighted aspects, the objectives of this study was: 1) To evaluate the effect of the particle used for air-abrasion on the surface roughness, wettability and morphological pattern of an yttria partially stabilized tetragonal zirconia, as well as to verify the correlation between the dependent variables roughness and wettability; 2) To evaluate the effect of air-abrasion moment on the yttria partially stabilized tetragonal zirconia surface characterization (roughness, morphology and phase transformation) and flexural strength, its shear bond strength to a resin cement and the predominant failure mode in the adhesive area. The correlation between the dependent variables roughness and bond strength was also verified. In the first study, specimens (14 × 14 × 1.4 mm) were obtained from Lava (3M ESPE AG) and, except for the control group (n=20), air-abraded with one of the following particles (n=20): 1) 50 μm Al_2O_3; 2) 120 μm Al_2O_3; 3) 250 μm Al_2O_3; 4) 30 μm silica-coated Al_2O_3 (Rocatec Soft); 5) 110 μm silica-coated Al_2O_3 (Rocatec Plus); 6) 120 μm Al_2O_3 and Rocatec Plus. The roughness (Ra) and wettability (by the silane RelyX Ceramic Primer) analyses were performed on the same specimens. For the surface morphology analysis by scanning electron microscopy (SEM), two additional specimens (6.0 × 6.0 × 1.0 mm) per group were prepared. The Ra (μm) data were analyzed by one-way ANOVA and Dunnett C test (α=0.05), and the wettability data by one-way ANOVA (α=0.05). Spearman correlation analysis was applied to test for a possible correlation between roughness and wettability. With the exception of the groups abraded with 120 μm Al_2O_3, the other ones presented statistically different

roughness values. Concerning wettability, there was no significant difference among the groups. No correlation (r_s=-0.09, *P*=0.27) was found between roughness and wettability. The particle influenced the surface morphology of the zirconia. The roughness increase seemed to have followed the size enlargement of the particles. The SEM analysis indicated that different particles provided differences in the morphological patterns. In the second study, specimens from Lava zirconia were air-abraded with 50 μm Al_2O_3 particles after (AS); before (BS); or before and after (BAS) zirconia sintering. For roughness (Ra), thirty specimens (10 × 10 × 3.0 mm) (n=10) were analyzed by a profilometer. Composite resin discs (n=30) were bonded with RelyX ARC (3M ESPE) on these specimens and stored in distilled water at 37 ºC for 24 h before the shear bond strength (SBS) test. Failure mode was determined with a stereomicroscope (×20). The surface morphology (n=2) was evaluated by SEM (×500). For the 4-point flexural strength (FS) test (EMIC DL2000), thirty-nine bar-shaped specimens (20 × 4.0 × 1.2 mm) (n=13) were air-abraded according to the three moments proposed, and an additional group (non-abraded) was evaluated (n=13). The phase transformation (n=1) was analyzed by Rietveld refinement with X-ray diffraction data. The Ra (μm) and SBS (MPa) data were analyzed by one-way ANOVA and Tukey's test (α=0.05). Pearson correlation analysis determined if there was correlation between roughness and SBS. For FS (MPa) data, one-way ANOVA and Dunnett C test (α=0.05) were used. Air-abrasion moment was significant (*P*<0.001) for Ra, SBS and FS data. The BS and AS groups presented the highest (1.3 μm) and the lowest (0.7 μm) Ra. The highest SBS (7.0 MPa) was observed for the BAS group, followed by the AS group (5.4 MPa) and, finally, by the BS group (2.6 MPa). All groups presented 100% adhesive failure. A weak correlation (r=-0.45,

P<0.05) was found between roughness and SBS. The surface morphology was influenced by the air-abrasion moment. The non-abraded (926.8 MPa) and BS (816.3 MPa) groups exhibited statistically similar FS values, but lower values than the AS (1249.1 MPa) and BAS (1181.4 MPa) groups, with no significant difference between them. The non-abraded, AS, BS, and BAS groups exhibited, respectively, percentages in weight of monoclinic phase of 0.0%, 12.2%, 0.0%, and 8.6%. The very rough surface provided by the air-abrasion before zirconia sintering impaired the bonding with the resin cement. The morphological patterns after the different air-abrasion moments were consistent with the surface roughness. Considering the short-term SBS and FS, the BAS group exhibited the best performance. Air-abrasion, regardless of its performance moment, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

Keywords: Ceramics, surface properties, shear strength, material resistance.

SUMÁRIO

Introdução

1 INTRODUÇÃO

Na Odontologia, o interesse pelas zircônias parcialmente estabilizadas por ítria aumentou consideravelmente nos últimos anos, devido às suas atrativas propriedades biológicas (ótima biocompatibilidade[32]), físicas (propriedades ópticas[7]), químicas (estabilidade química[3]) e mecânicas (maiores tenacidade[56,60,74] e resistência à fratura[10,38,69,74] dentre as cerâmicas dentais). Atualmente, as excelentes propriedades mecânicas das zircônias permitem que este material seja utilizado na confecção de restaurações extensas e localizadas em regiões de grande esforço mastigatório[10,38,69,74].

Para que estas restaurações, assim como as confeccionadas em outros materiais, tenham boa qualidade, além de longevidade, é necessário que todos os passos de sua confecção sejam executados com rigor, para que, ao final, a soma de erros seja a menor possível. Uma das etapas, tão importante quanto as demais, é a cimentação[16]. No caso das restaurações com infraestrutura de zircônia, para o favorecimento da retenção mecânica e da longevidade clínica, os cimentos resinosos são os mais recomendados[58], por apresentarem propriedades mecânicas melhoradas em relação aos demais cimentos[57,62] e pela possibilidade de se ter uma cimentação adesiva com eles[61,71].

No âmbito da cimentação, duas interfaces devem ser avaliadas: 1) interface cimento/dente e 2) interface cimento/restauração. Considerando esta última interface, que é o foco do presente estudo, dois mecanismos de união podem existir: mecânico (retenção) e químico (adesão). De acordo com alguns autores[12,30,33,58,68], para que a cimentação seja bem sucedida, é importante que

estejam presentes ambos os mecanismos de união. O mecânico ocorre por meio da imbricação do cimento nas retenções micromecânicas presentes na superfície da restauração, enquanto o químico é estabelecido por reações ocorridas entre determinados componentes do cimento e alguns elementos presentes na superfície do material restaurador. Estudos[67,75,77,79] indicam que, sem a presença do mecanismo mecânico, ou seja, se não houver microrretenções na superfície do material restaurador, o mecanismo químico torna-se ineficaz, com valores de resistência adesiva incompatíveis com a necessidade clínica. Por outro lado, o mecanismo mecânico, quando isolado, promove uma resistência adesiva que poderia ser melhorada com a associação do mecanismo químico[30,37,43,67,79]. Alguns autores[59,78] ainda afirmam que a contaminação da superfície interna das infraestruturas de zircônia, durante o seu manuseio, pode prejudicar a união química, enquanto outros[4,54] chamam a atenção para a susceptibilidade das ligações químicas estabelecidas na interface com relação à degradação hidrolítica.

Nos sistemas cerâmicos que contém elevado conteúdo de matriz vítrea, o mecanismo mecânico da união pode ser obtido por meio do condicionamento com ácido fluorídrico (HF) 2,5-10%, que ataca essa matriz criando irregularidades na superfície[9,11]. Já com relação às cerâmicas com elevado conteúdo cristalino e matriz vítrea limitada (abaixo de 1%)[55], como é o caso das zircônias (Lava, Cercon, Zirkonzahn, Procera Zirconia), o condicionamento com HF não é eficaz[23,35]. Portanto, devem ser empregados tratamentos de superfície alternativos que consigam criar microrretenções na superfície, que favoreçam a união mecânica. Dentre eles, o jateamento é o mais simples e o mais utilizado[14,16,21,65-66,75], apesar de existirem outros métodos como

a aplicação de laser[16,66], deposição de alumina[31], deposição de sílica[19], deposição de porcelana feldspática[24], *selective infiltration etching (SIE)*[2,14], *hot etching solution*[14], entre outros. Além de criar rugosidade, o jateamento limpa a superfície e aumenta a área disponível para união, favorecendo a imbricação mecânica do cimento na superfície da restauração[16,75,77] e, também, aumentando a energia livre de superfície, o que irá permitir um maior molhamento da superfície pelo material aplicado na sequência.[9,76]

Duas composições diferentes de partículas podem ser utilizadas no jateamento: as convencionais e as revestidas. As convencionais são constituídas simplesmente por óxido de alumínio/alumina (Al_2O_3), enquanto as revestidas são partículas de Al_2O_3 com revestimento por sílica. Ambas as partículas podem ser encontradas em diversos tamanhos. As partículas convencionais geralmente estão disponíveis nos tamanhos de 25 µm a 250 µm[17,26,67,70]. As partículas revestidas, com seus respectivos tamanhos, estão presentes nos sistemas Cojet Sand[5,26]/Rocatec Soft (30 µm)[67,76] e Rocatec Plus (110 µm)[5,26,76], ambos da 3M ESPE, bem como em outros sistemas disponíveis no mercado europeu como as partículas de 50 µm fabricadas pela Supradental (Madri, Espanha)[54-55]. Estas partículas revestidas, além de favorecerem a retenção na interface cimento/restauração, depositam uma camada de sílica na superfície da zircônia[6,15,41,46]. Portanto, o jateamento realizado com essas partículas cria união mecânica e prepara a superfície para uma união química que se consagra com a aplicação do silano realizada após o jateamento[5,33,68].

Apesar da importância da existência do mecanismo mecânico na interface cimento/restauração, que geralmente é criado pelo jateamento, e da ampla gama de opções de partículas de Al_2O_3 utilizadas em tal procedimento, as

informações que existem na literatura a respeito dos efeitos da variabilidade de tamanho na morfologia, na rugosidade e no molhamento da zircônia jateada, propriedades estas fundamentais no mecanismo mecânico de união, são escassas e controversas.

Quanto à morfologia de superfície, alguns estudos[48,55,70,73] avaliaram os efeitos do jateamento com diferentes tamanhos de partículas na superfície da zircônia. Oyagüe et al.[55] observaram que o jateamento da zircônia com partículas de Al_2O_3 revestidas por sílica de 50 µm promoveu apenas uma pequena alteração na textura de superfície em relação ao grupo controle (sem tratamento), enquanto o jateamento com partículas de Al_2O_3 de 125 µm produziu microrretenções na forma de "lâmina de faca", sem fendas. Outros autores[73] também constataram que o jateamento com partículas de Al_2O_3 de maior tamanho (120 µm) promoveu mudanças no padrão morfológico de superfície (trincas e arranhões profundos, remoção de grãos e perda de material) mais significativas do que o jateamento com partículas menores (50 µm). Monaco et al.[48], por meio de microscopia eletrônica de varredura, verificaram que o jateamento com partículas de Al_2O_3 de 50 µm e 110 µm, proporcionalmente ao tamanho da partícula, resultou em uma superfície deformada plasticamente, com trincas e remoção da camada superficial, enquanto a superfície jateada com partículas de Al_2O_3 de 30 µm revestidas por sílica, apresentou pequenas trincas. No estudo de Turp et al.[70], observou-se que com o aumento do tamanho das partículas de Al_2O_3 (30 µm, 50 µm, 110 µm e 250 µm) a morfologia de superfície apresentou um aspecto de "achatamento". Casucci et al.[13] avaliaram somente um tamanho de partículas de Al_2O_3 (125 µm) e verificaram que os grupos jateados com elas, bem como aqueles condicionados com HF 9,5% por 90

segundos apresentaram o mesmo padrão de morfologia de superfície quando comparados ao grupo que não recebeu tratamento (grupo controle).

Com relação à rugosidade, embora diferentes valores sejam encontrados na literatura, os estudos[18,21,48,50-51,53,55,65-66,73,76] apontam para a influência do jateamento no aumento da rugosidade da superfície da zircônia, sendo que partículas maiores tendem a promover maior rugosidade[48,50,55,65-66,70,73,76]. Apesar disso, Curtis et al.[20] observaram que esse procedimento realizado com partículas de Al_2O_3 de 25 µm, 50 µm ou 110 µm reduziu a rugosidade da zircônia Lava quando comparado ao grupo controle (que representa os espécimes como vêm do laboratório), não tendo havido diferença significativa na atuação dos três tamanhos de partícula. Estes autores justificaram seus resultados como uma possível redução de defeitos existentes na superfície da zircônia inerentes ao material ou resultantes de seu processamento. Possivelmente estas diferenças nos valores de rugosidade encontradas nos estudos são resultantes dos diferentes métodos de análise (rugosímetro[20-21,48,50-51,65-66,73,76], microscopia de força atômica[55], microscopia confocal[18], interferometria óptica[53,70]), de obtenção e preparo dos espécimes, bem como das diferenças relacionadas ao tamanho do grão, composição, densidade e dureza das zircônias comercialmente disponíveis[18].

As zircônias parcialmente estabilizadas por ítria, por apresentarem inércia química[63,68] e superfície homogênea[33,63], necessitam de tratamentos capazes de aumentar o molhamento e a energia de superfície, favorecendo a união com materiais resinosos. Entretanto, embora alguns estudos[72,76] tenham observado que o jateamento aumenta a energia livre de superfície, o que permitiria maior molhamento da superfície, somente Noro et al.[50] estudaram o

efeito da variabilidade de tamanho (25 µm, 50 µm e 150 µm) das partículas de Al_2O_3 convencionais nessa propriedade, por meio da utilização de água destilada. Portanto, apesar da importância do molhamento na questão da resistência adesiva da interface zircônia/cimento[9], os efeitos do tamanho (de 25 µm a 250 µm) e da composição (convencionais e revestidas) das partículas empregadas no jateamento nesta propriedade precisam ser melhor elucidados. Geralmente, após o jateamento da superfície da zircônia, independentemente do tamanho e do tipo de partícula utilizados, é aplicado um silano cujo objetivo secundário, não menos importante que o primário e que será apresentado mais adiante, é o de promover um aumento do molhamento da zircônia pelo cimento[8-9,37,44-46,68]. Para que isto ocorra da melhor forma possível, é importante que haja um molhamento adequado, pelo próprio silano, da zircônia jateada. Portanto, a avaliação do molhamento da superfície da zircônia pelo silano mediante diferentes tratamentos de superfície é imprescindível.

Retornando à função principal dos silanos, estes devem estabelecer união química entre a zircônia e o cimento pelas seguintes possíveis reações, que ocorrem concomitantemente: 1) entre a porção silicofuncional do silano e a zircônia (óxido de zircônio)[46,52]; 2) entre a sílica e/ou a alumina depositadas na superfície da zircônia (dependendo da partícula empregada no jateamento) e a porção silicofuncional do silano[5,19,42,46]; e 3) entre os monômeros resinosos do cimento e a porção organofuncional deste agente de união[5,19,42,45,68].

Dependendo da modalidade de jateamento, podem estar presentes no substrato: 1) zircônia + alumina (no jateamento com partículas de Al_2O_3 convencionais)[41] ou 2) zircônia + alumina + sílica (no jateamento com

partículas de Al_2O_3 revestidas e nos sistemas que empregam jateamento com partículas de Al_2O_3 convencionais seguido do jateamento com partículas de Al_2O_3 revestidass)[15,41,46]. Portanto, nas duas situações apresentadas acima, os compostos zircônia, alumina e sílica que, provavelmente, possuem diferentes graus de afinidade e estabelecem diferentes forças de reação com a porção silicofuncional do silano, concorrem na reação com este agente de união. Alguns estudos[5,34,42,45-46] indicam que as ligações químicas estabelecidas entre o silano e a sílica resultante do jateamento com partículas de Al_2O_3 revestidas (≡Si-O-Si≡) são mais fortes e hidroliticamente estáveis do que aquelas entre o silano e a alumina ocorridas quando do emprego de partículas de Al_2O_3 convencionais (=Al-O-Si≡). Outros comentam sobre a existência de uma relação fraca entre a zircônia e o silano (≡Zr-O-Si≡)[8,34,42,46,68].

Uma outra preocupação é que, as zircônias sinterizadas, por serem materiais compactos[33], apresentando elevada dureza[17,22,68,74], podem oferecer resistência ao jateamento[13], necessitando, portanto, que, nesse procedimento, seja empregada uma pressão mais elevada ou partículas maiores (com relação às empregadas no jateamento de ligas metálicas) para a criação de microrrugosidades na superfície jateada, necessárias para que ocorra a imbricação na interface restauração/cimento. Contudo, o uso de partículas maiores pode criar defeitos na superfície da zircônia, prejudicando sua interface com o cimento[52].

Além disso, embora alguns autores tenham mostrado que, em curto prazo, o jateamento pode até promover um aumento da resistência mecânica da zircônia[27,39-40,58,64], Fonseca et al.[26] e Turp et al.[70] observaram que o comportamento deste material depende do grau de severidade do jateamento,

sendo que partículas maiores, como as de 250 µm, podem causar danos ao material. Além do tamanho das partículas, sua composição, bem como a pressão e o tempo empregados no jateamento são fatores que vão influenciar a resposta da zircônia. Zhang et al.[80] sugerem o emprego de microesferas de sílica, que são mais macias e mais arredondadas do que as partículas de alumina. Além do tamanho das partículas, Turp et al.[70] concluíram que tempos maiores de jateamento podem levar à degradação do material.

O jateamento cria defeitos e microtrincas na superfície da zircônia, em quantidade e profundidade variadas, dependendo da severidade do procedimento. Ao redor dessas, ocorrerá transformação de fase (tetragonal para monoclínica) acompanhada por aumento volumétrico em torno de 5%[29] que, por sua vez, vai gerar forças compressivas, as quais conseguirão ou não conter a propagação destas trincas em direção ao corpo do material, promovendo, respectivamente, aumento[26-27,39-40,58,64] ou redução[1,26,28,36] da resistência mecânica da zircônia.

Com o passar do tempo, a atuação da fadiga mecânica propiciará a propagação daquelas trincas resultantes do jateamento. Zhang et al.[80] reportaram que, após fadiga cíclica correspondente a 1 ano de contatos oclusais, a redução da resistência da zircônia jateada (partículas de Al_2O_3 de 50 µm, 0,276 MPa, 5 segundos, distância de 10 mm) foi de 20% a 30% maior que a redução da resistência da zircônia somente polida. Portanto, o jateamento associado à fadiga mecânica, pode acelerar a perda da resistência, pela propagação das microtrincas pelo corpo do material. Além disto, Kim et al.[36] comentam que as microtrincas facilitam o processo conhecido como *low temperature degradation (LTD),* pois promovem uma passagem para a água,

quando da presença de umidade. Portanto, também em longo prazo, quanto mais severo tiver sido o jateamento, microtrincas em maior número e mais profundas irão existir, tornando a zircônia mais susceptível à redução de sua resistência.

Zhang et al.[80] comentam que embora as forças compressivas resultantes da transformação de fase possam conter as microtrincas, com a sinterização da porcelana de revestimento, o calor gerado promoveria uma liberação/relaxamento destas forças, as quais deixariam de impedir a propagação das microtrincas, resultando em redução da resistência mecânica da zircônia. Essa consideração, nos leva a pensar que o momento em que o jateamento é realizado também é um fator que pode vir a influenciar a resistência da zircônia. Geralmente, o jateamento é realizado na zircônia sinterizada. Entretanto, se o realizássemos antes da sinterização deste material, as microtrincas resultantes do jateamento poderiam ser "seladas" ou "contidas" pela contração de sinterização da zircônia[36], geralmente em torno de 20% a 25%. Além disto, com a sinterização da zircônia, o conteúdo de fase monoclínica (gerada pelo jateamento) praticamente zeraria[48-49] e, quando da sinterização da porcelana de revestimento, não haveria transformação de fase reversa (monoclínica para tetragonal) com consequente cessação das forças compressivas e, portanto, hipoteticamente, não haveria redução da resistência. Talvez, uma simples mudança no momento em que o jateamento é realizado, permitisse a utilização de partículas menores e menor pressão, possibilitando a anulação dos efeitos da severidade do jateamento e modificando o comportamento mecânico da zircônia, tanto em curto quanto em longo prazo.

Na literatura, encontramos poucos estudos que avaliaram o efeito do momento do jateamento e, em nenhum deles, a resistência mecânica da zircônia foi avaliada. Monaco et al.[48] observaram que a superfície da zircônia jateada com partículas de Al_2O_3 de 50 µm antes da sinterização apresentou maior rugosidade (2,33 µm) em relação àquela jateada após a sinterização (0,48 µm), a qual exibiu uma deformação plástica. Moon et al.[49], por sua vez, concluíram que a superfície da zircônia jateada previamente à sinterização apresentou aspecto morfológico mais arredondado, isto é, com arestas não tão evidenciadas, enquanto a superfície sinterizada, quando jateada, apresentou padrão "em forma de agulha", bastante irregular e com arestas bem evidentes. Com relação à resistência de união entre zircônia e materiais resinosos, Fazi et al.[25], Monaco et al.[47] e Moon et al.[49] não encontraram diferença significante no jateamento realizado antes a após a sinterização da zircônia. Além destas propriedades, a análise de transformação de fase pode ser um instrumento complementar para indicar como os diferentes momentos do jateamento podem ser agressivos ou não à zircônia. Monaco et al.[48], após o jateamento com partículas de Al_2O_3 de 30 µm revestidas por sílica e partículas de Al_2O_3 de 50 µm e 110 µm, observaram que a superfície da zircônia apresentou conteúdo de fase monoclínica de 10%, 8% e 14% em massa, respectivamente. No entanto, esses autores[48] verificaram que a zircônia jateada previamente à sua sinterização, quando sinterizada, apresentou conteúdo de fase monoclínica em torno de 0% em massa. Esse mesmo comportamento foi observado por Moon et al.[49], os quais concluíram que o jateamento da zircônia sinterizada resultou em 11,4% em massa de fase monoclínica, enquanto a superfície pré-sinterizada, quando jateada, apresentou conteúdo de 16,9% em massa dessa mesma fase, o qual

praticamente foi zerado após a sinterização. Devido à contração de sinterização da zircônia (em torno de 20%), esses autores[49] utilizaram partículas de Al_2O_3 de tamanhos diferentes para o jateamento pré- (70 μm) e pós-sinterização (50 μm), com o intuito de obter características de superfície similares entre os grupos experimentais.

Diante de todos os fatos abordados e da ampla gama de estudos sobre zircônia, muitas dúvidas ainda permanecem em relação aos efeitos das partículas empregadas no jateamento, mais especificamente do seu tamanho e, em algumas situações também da sua composição, na caracterização de superfície (morfologia, rugosidade, molhamento) da zircônia. Todas essas informações são imprescindíveis para uma maior compreensão das interações e comportamentos envolvidos na interface zircônia/cimento. Além disso, a utilização do jateamento, que se constitui em um método tão simples, em diferentes momentos (antes ou após a sinterização da zircônia), pode propiciar a obtenção da rugosidade necessária para que ocorra a imbricação mecânica na interface zircônia/cimento, sem prejuízo da resistência mecânica da zircônia.

Proposição

2 PROPOSIÇÃO

Os objetivos deste estudo in vitro foram:

1) Avaliar o efeito da partícula empregada no jateamento na rugosidade de superfície, molhamento e padrão morfológico de uma zircônia parcialmente estabilizada por ítria, bem como verificar a existência de correlação entre as variáveis dependentes rugosidade e molhamento.

2) Avaliar o efeito do momento do jateamento na caracterização de superfície (rugosidade, morfologia e transformação de fase) e resistência à flexão de uma zircônia parcialmente estabilizada por ítria, bem como sua resistência de união ao cisalhamento com um cimento resinoso e o modo de fratura predominante na área adesiva. A existência de correlação entre as variáveis dependentes rugosidade e resistência de união também foi verificada.

Cada um dos objetivos específicos acima está apresentado em 1 capítulo na forma de artigo científico:

Capítulo 1: *"Evaluation of roughness, wettability and morphology of a Y-TZP ceramic after different air-abrasion protocols"*

Capítulo 2: *"The effect of different air-abrasion moments on the Y-TZP ceramic surface characterization, flexural strength and shear bond strength to resin cement"*

Capítulos

3.1 CAPÍTULO 1

Evaluation of roughness, wettability and morphology of a Y-TZP ceramic after different air-abrasion protocols*

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ABSTRACT

Statement of problem: Airborne-particle abrasion is an effective method for roughening the zirconia surface and promoting micromechanical interlocks with luting cements. However, the effect of different air-abrasion protocols on aspects strictly related to the micromechanical retention mechanism has been poorly investigated.

Purpose: To evaluate the effect of air-abrasion protocols on the surface roughness, wettability and morphology of a Y-TZP ceramic.

Material and Methods: One hundred and forty zirconia specimens (14 × 14 × 1.4 mm) were made from Lava (3M ESPE AG) and their surfaces were randomly treated as follows (n=20): 1) as-sintered (control); 2) airborne-particle abraded with 50 μm Al_2O_3 particles; 3) 120 μm Al_2O_3 particles; 4) 250 μm Al_2O_3 particles; 5) 30 μm silica-coated Al_2O_3 particles (Rocatec Soft); 6) 110 μm silica-coated Al_2O_3 particles (Rocatec Plus); 7) 120 μm Al_2O_3 particles followed by Rocatec Plus. The surface roughness (Ra) and wettability (by the silane coupling agent RelyX Ceramic Primer) analyses were performed on the same specimens of each group. Two additional specimens (6.0 × 6.0 × 1.0 mm) per group were prepared to evaluate the surface morphology using SEM (scanning electron microscopy). Roughness (Ra) data were analyzed by one-way ANOVA and Dunnett C test (α=.05). The analysis of wettability data was performed by one-way ANOVA (α=.05). Spearman correlation analysis was applied to test for a possible correlation between roughness and wettability.

Results: The control group (0.35 μm) exhibited the lowest mean roughness value (Ra), which was followed by Rocatec Soft (0.40 μm), 50 μm Al_2O_3 particles (0.52 μm), Rocatec Plus (0.69 μm), 120 μm Al_2O_3 particles (0.80 μm) and 120 μm

Al_2O_3 particles + Rocatec Plus (0.79 μm), and finally by 250 μm Al_2O_3 particles (1.13 μm), which provided the highest roughness. There was no significant difference among the groups concerning wettability. No correlation (r_s=-0.09, *P*=.27) was found between the two dependent variables. Different air-abrasion protocols influenced the zirconia morphology.

Conclusions: The roughness increase seemed to have followed the size enlargement of the particles. The SEM analysis indicated that different air-abrasion protocols provided differences in the morphological patterns.

CLINICAL IMPLICATIONS

Although the airborne-particle abrasion with larger particles provides rougher surfaces, when considering roughness, wettability and morphology together, abrasion only with 120 μm Al_2O_3 particles seems to be the most promising procedure regarding the mechanical bonding mechanism. The wettability of the zirconia surface by the RelyX Ceramic Primer silane is not influenced by particle size and surface roughness. Rocatec Plus is not necessary after abrasion with 120 μm Al_2O_3 particles.

Keywords: zirconia, airborne-particle abrasion, surface roughness, wettability, morphology

INTRODUCTION

For a long-term durable bond at the zirconia/cement interface, the surface of Y-TZP ceramic restorations should be able to promote micromechanical interlocking and establish chemical reactions with resin cements.[1,2] Some authors[3,4] highlight the essential role of the micromechanical retention to improve the bonding with luting cements. Others claim that the contamination of the inner surface of the zirconia frameworks, during handling, can affect chemical bonding,[5,6] or that the materials at the bonding interface are susceptible to hydrolytic degradation.[7,8]

The micromechanical bonding mechanism is a result of cement infiltration into the micro-irregularities on the inner surface of the restoration. Although there are some alternative surface treatments that provide mechanical bond, airborne-particle abrasion is one of the most used and studied methods for zirconia.[3,9-16] Besides roughening the surface, this procedure cleans it and increases available surface area for bonding, favoring the mechanical interlocking between the cement and the airborne-particle abraded zirconia surface,[3,10,17] and also increasing the surface free energy, which enhances the wettability of the substrate by the material applied afterwards.[18,19]

For airborne-particle abrasion there is a variety of particle size (25 μm to 250 μm) and composition (conventional alumina particles or silica-coated ones).[20,21] Air-abrasion with silica-coated Al_2O_3 particles, besides favoring the micromechanical retention at the zirconia/cement interface, deposits a silica layer on the zirconia surface,[22,23] preparing it to react with the silane (hydrolyzable alkoxy groups) applied afterwards.[24,25] However, despite the importance of the airborne-particle abrasion and the wide range of particle options used in this

procedure, the information found in the literature about the effects of particle size on roughness,[19,26-30] morphology,[26,27,29,30] and wettability[31] of the zirconia surface are controversial or scarce.

Thus, the purpose of this in vitro study was to evaluate the effect of air-abrasion protocols on surface roughness, wettability (dependent variables) and morphology of a Y-TZP ceramic. The null hypotheses determined that the air-abrasion protocol does not influence the dependent variables, and that there is no correlation between them.

MATERIAL AND METHODS

Preparation and surface treatment of zirconia specimens

The materials evaluated in this study are summarized in Table I.

One hundred and forty pre-sintered zirconia specimens were obtained from Lava frames (3M ESPE AG), which were cut with a saw (Isomet 1000; Buehler Ltd.) under water irrigation. The specimens were washed in tap water to remove the cutting debris and finished manually using a ceramic polisher (Exa Cerapol 0361HP; Edenta AG) in a slow-speed handpiece. One surface of each specimen was polished with 600- and 1200-grit silicon carbide abrasive papers under wet conditions.

After sintering in a specific oven (Lava Furnace 200; Dekema Dental-Keramiköfen GmbH) according to the manufacturer's instructions (heating rate=20 ºC/min: 0 ºC-1000 ºC; 10 ºC/min: 1000 ºC-1500 ºC; holding time=2 h and cooling rate=15 ºC/min: 1500 ºC-800 ºC; 20 ºC/min: 800 ºC-250 ºC – the oven was opened at 250 ºC), all zirconia specimens (14 × 14 × 1.4 mm) were subjected to one of the following surface treatment conditions (n=20): 1) as-

sintered (control); 2) airborne-particle abraded with 50 μm Al_2O_3 particles; 3) 120 μm Al_2O_3 particles; 4) 250 μm Al_2O_3 particles; 5) 30 μm silica-coated Al_2O_3 particles (Rocatec Soft); 6) 110 μm silica-coated Al_2O_3 particles (Rocatec Plus); 7) 120 μm Al_2O_3 particles followed by Rocatec Plus (similarly to the recommendation of the manufacturer of the Rocatec Plus).

For the airborne-particle abrasion procedure, the specimens were mounted on a special holder and airborne-particle abraded for 15 s with an air-abrasion unit (Basic Classic; Renfert GmbH) at a pressure of 0.28 MPa and a perpendicular distance of 10 mm from the zirconia surface.[24] All specimens were cleaned in 99% isopropanol for 10 min in ultrasonic bath, and left to dry at room temperature for 24 h.

Surface roughness measurements

Prior to contact angle measurements, the average surface roughness (Ra in μm) of all specimens was determined using a profilometer (Surftest SJ-400; Mitutoyo Corporation) with a cut-off value (λ_c) of 0.8 mm.[12,16,19] Resolution was 0.01 mm, the transverse length (length of the surface examined by the instrument) was 2.4 mm, and the speed of the diamond stylus with a 5-μm tip radius was 0.5 mm/s. Three equidistant parallel measurements were made with a perpendicular stylus on different areas of the specimen. All measurements were performed perpendicular to the direction of the airborne-particle abrasion. The average reading was designated as the Ra value of each specimen. One calibrated operator (Intraclass Correlation Coefficient - ICC=0.89, Tabelas A1 e A2) recorded all measurements.

Contact angle measurements

Contact angle is defined as the angle at the intercept of a plane tangent to a drop and the plane containing the substrate-liquid interface. The wettability of the zirconia surface by the silane RelyX Ceramic Primer was characterized by means of the contact angle formed between the silane and the airborne-particle abraded zirconia surface. For the measurements, an automated goniometer (model 200-00; Ramé-Hart Instrument Co.) comprising a CCD camera was used to record the image of a liquid drop placed onto the surface by a microsyringe, while an image processing software determined the contact angle. One calibrated operator (ICC=0.67, Tabelas A1 e A2) obtained two measurements in each specimen and the average was determined.

Statistical analysis

For roughness, since the homogeneity assumption was violated (Levene, P=.00), the data were analyzed by one-way analysis of variance (ANOVA) and Dunnett C test (α=.05). For wettability, the assumptions of the analysis were satisfied (Shapiro-Wilk, .18≤P≤.87; Levene, P=.08), thus the data were submitted to one-way ANOVA (α=.05) (IBM SPSS Statistics version 20; Statistical Package for Statistical Science Inc.). To estimate the significance of the correlation between roughness and wettability, the Spearman rank correlation test was used.

Scanning electron microscopy analysis

The scanning electron microscopy (SEM) analysis was performed to characterize the morphological patterns of the zirconia surface after the

different airborne-particle abrasion protocols. Two additional specimens (6.0 × 6.0 × 1.0 mm) from each experimental group were prepared, mounted on metallic stubs and analyzed under a high-resolution field emission scanning electron microscope (model JSM-7500F; JEOL Ltd.) at ×500 magnification and an accelerating voltage of 2.0 kV.

RESULTS

The one-way ANOVA results showed that the air-abrasion protocol was significant for roughness (*df*=6, F=348.1, *P*=.00) and did not influence the wettability (*df*=6, F=0.7, *P*=.66).

Table II (Tabelas A3 e A4) shows the mean Ra (μm) values, standard deviations and statistical analysis results identified with Dunnett C test, as well as the contact angle (°) mean values and standard deviations. The control group (0.35 μm) exhibited the lowest mean roughness value (Ra), which was followed by Rocatec Soft (0.40 μm), 50 μm Al_2O_3 particles (0.52 μm), Rocatec Plus (0.69 μm), 120 μm Al_2O_3 particles (0.80 μm) and 120 μm Al_2O_3 particles + Rocatec Plus (0.79 μm), and finally by 250 μm Al_2O_3 particles (1.13 μm), which provided the highest roughness. There was no significant difference in wettability among the groups.

For the correlation analysis between roughness and wettability, Spearman's correlation test revealed no correlation (r_s=-0.09, *P*=.27) between the two variables.

The representative SEM images (Figure 1) indicated that different air-abrasion protocols provided differences in the morphological patterns of the zirconia surface.

DISCUSSION

The results of this study did not find evidence to support the null hypothesis, as the particle size affected the zirconia surface roughness, as well as morphology. The influence of the particles on roughness and/or morphology had already been observed in other studies, both in metal[32,33] and zirconia.[19,26-30]

In the present study, air-abrasion with 30 μm silica-coated Al_2O_3 particles (Rocatec Soft) provided significantly higher roughness than the as-sintered group (control), as observed in other studies.[15,16,19,22,27] Although the particles of Rocatec Soft (30 μm) are smaller than the others, they were able to rough the zirconia surface in comparison with the control group, as observed by SEM analysis, which revealed that the control group and that abraded with Rocatec Soft apparently presented a surface that was less and more favorable to the micromechanical retention. This difference in morphology was also observed by Subaşı and İnan.[15,16] The mean roughness value (Ra) found in the present study for the as-sintered group is consistent with the studies of Monaco et al[27] and Cavalcanti et al,[11] while the roughness of the Rocatec Soft group is corroborated by the study of Cattani Lorente et al.[22]

The significantly higher roughness provided by 50 μm Al_2O_3 particles in comparison with the control group and/or that abraded with 30 μm silica-coated Al_2O_3 particles was also found in the literature.[11,14,27-30] Some authors observed that the abrasion with 50 μm[14,30] or 53 μm[11] Al_2O_3 particles presented higher roughness than the control group. Monaco et al[27] and Özcan et al[28] concluded that 50 μm Al_2O_3 particles provided a rougher surface in comparison with the nonabraded group and that abraded with 30 μm silica-coated Al_2O_3 particles. Other researchers[29] observed higher roughness with 50

μm Al_2O_3 particles in comparison with Cojet Sand. On the other hand, Yamaguchi et al[19] reported that 70 μm Al_2O_3 particles promoted significantly higher roughness than the control group and that abraded with Rocatec Soft. Concerning the morphologic analysis, we observed that, differently from the control group, which exhibited a smooth surface, abrasion with Rocatec Soft and 50 μm Al_2O_3 particles resulted in a change in surface texture, forming more evident microretentive grooves with the latter particles. Monaco et al[27] reported the same behavior when these three conditions were evaluated (control group, Rocatec Soft and 50 μm Al_2O_3 particles). Oguri et al[14] and Cavalcanti et al[11] observed the same behavior, respectively, with 50 μm and 53 μm Al_2O_3 particles in comparison with the control group. Contrary to these findings, Foxton et al[13] reported that air-abrasion with 53 μm Al_2O_3 particles did not appear to significantly change the zirconia surface appearance in comparison with the control group.

The Rocatec Plus group exhibited the third highest roughness. When this group was compared with that abraded with Rocatec Soft (30 μm) or 50 μm Al_2O_3 particles, it was observed a significant increase in the surface roughness mean value. This behavior may be attributed to the higher grain size (110 μm) of the Rocatec Plus particles. Monaco et al[27] verified the effect of the particle size on roughness and morphology and observed that the larger Al_2O_3 particles (110 μm) provided higher roughness and more marked deformations than the smaller silica-coated (30 μm) and alumina (50 μm) ones. Turp et al,[29] in turn, also concluded that the abrasion with larger alumina particles (110 μm) resulted in a rougher surface in comparison to that one obtained after abrasion with Cojet Sand (30 μm) or 50 μm Al_2O_3 particles. Concerning the silica-coated

Al_2O_3 particles, Yamaguchi et al[19] observed that the 110 μm particles provided higher roughness than the 30 μm ones.

The groups abraded with 120 μm Al_2O_3 particles and 120 μm Al_2O_3 particles + Rocatec Plus provided the second highest roughness value. Regarding the superiority of these groups over the 50 μm Al_2O_3 particles, Monaco et al,[27] Turp et al[29] and Wang et al[30] also reported significantly higher roughness after abrasion with 110 μm and 120 μm Al_2O_3 particles in comparison with that of the group abraded with 50 μm ones. Only Curtis et al[26] observed statistically similar roughness values between the groups abraded with 50 μm and 110 μm Al_2O_3 particles. The higher roughness provided by the larger particles (110 μm/120 μm) with respect to the 50 μm ones was corroborated by the scanning electron microscopy performed in this study. The SEM analysis indicated different morphological patterns, with advantages for the 110 μm and 120 μm Al_2O_3 particles, which promoted a surface more favorable for micromechanical retention at the zirconia/cement interface.

Concerning the superiority of the 120 μm Al_2O_3 particles (0.80 μm) and 120 μm Al_2O_3 particles + Rocatec Plus (0.79 μm) over Rocatec Plus (0.69 μm), this comparison was not found in the literature. We can suppose that although the difference between the particle sizes is quite small (10 μm), it was enough for determining higher roughness when compared to that of the group abraded with the silica-coated ones. This fact also explains why the double airborne-particle abrasion, as recommended by the manufacturer (3M ESPE), provided no significant difference in roughness when compared to the group abraded only with 120 μm Al_2O_3 particles. Comparing the morphology in these three groups, the group abraded only with 120 μm Al_2O_3 particles seems to

provide a surface with more edge-shaped micro-irregularities than the other two groups, which exhibited a flatter pattern. Therefore, both roughness and morphology analyses indicate that in those three groups abrasion with 120 μm Al_2O_3 particles seems to be more effective in terms of micromechanical retention.

Different roughness values were found in the literature. For the group abraded with 50 μm Al_2O_3 particles, Monaco et al[27] and Oguri et al[14] reported values of around 0.48 μm, very close to that found in the current study (0.52 μm). However, Turp et al[29] and Noro et al[31] reported mean values of ~0.70 μm and 0.87 μm for the same particle size. Cavalcanti et al.[11], in turn, after abrasion of the Cercon and Procera zirconia surfaces with 53 μm Al_2O_3 particles, observed roughness values of 2.41 μm and 0.86 μm, respectively. For abrasion with Rocatec Plus, different from this study (0.69 μm), Özcan et al[28] and Yamaguchi et al[19] obtained 0.20 μm and 0.50 μm, respectively. Regarding abrasion with 120 μm Al_2O_3 particles, we observed 0.80 μm, while the study by Wang et al[30] showed 1.18 μm. Other studies[12,15,16] used 110 μm Al_2O_3 particles, whose size is very close to the 120 μm Al_2O_3 ones, and reported roughness values between 0.74 μm and 0.80 μm. It is very likely that these differences in roughness values found in the studies may be a result of the different analysis methods (profilometer,[12,14-16,19,27,30,31] optical interferometry,[28,29] confocal microscopy[11]) and different methods of manufacturing and preparation of the specimens, as well as of the differences related to the grain size, composition, density and hardness of the commercially available Y-TZP ceramics.[11]

With respect to the higher roughness (1.13 μm) provided by abrasion with 250 μm Al_2O_3 particles, some studies[27,30,34] also reported an increase in this property with particle size enlargement used for the airborne-

particle abrasion. In addition, these authors[27,30,34] also observed that the use of larger particles provided a clearly rougher profile than the smaller ones. However, these studies did not evaluate abrasion with 250 μm Al_2O_3 particles. Although, in the present study, this particle size promoted the highest roughness, the morphological analysis exhibits a flatter and broader pattern than that observed for 110 μm/120 μm particles. Another concern is that air-abrasion with this particle size may result in severe surface damage and material loss, which could compromise the mechanical properties of zirconia. A study by Fonseca et al[20] showed a decrease in flexural strength when zirconia was abraded with 250 μm Al_2O_3 particles and, contrastingly, an increase in this property with 50 μm and 110/120 μm Al_2O_3 particles.

Regarding the contact angle analysis, although an optimal wettability of the zirconia by the adhesive agent is essential for achieving a strong adhesion at the zirconia/resin cement interface, the effects of different factors capable of modifying the wettability of the zirconia surface were poorly investigated.[14,19,31,35,36] Among the studies, only that by Noro et al.[31] evaluated the effect of Al_2O_3 particle size used for airborne-particle abrasion on the zirconia wettability by distilled water. Although the authors[31] used a test liquid different from the present study, their results corroborate ours, since the particle size enlargement seemed not to influence the wettability of the abraded zirconia surface. Yamaguchi et al[19] investigated the effect of the particle size on the surface free energy of Lava zirconia, which is closely related to wettability. Changes in the surface energy of a substrate cause alterations in its wetting capability by a certain liquid.[36] Yamaguchi et al[19] observed the following descending order of mean surface free energy values: Rocatec Soft (30 μm),

Rocatec Plus (110 µm), 70 µm Al_2O_3 particles, and finally the control group. This result indicates that, regarding surface free energy, the airborne-particle abrasion with silica-coated Al_2O_3 particles is more favorable. However, in terms of wettability this behavior was not observed in the present study since there was no significant difference in the contact angle among all groups. The novelty of the current research regards that, besides the effect of different particle sizes on wettability, the test liquid used for contact angle analysis was a silane, which is a highly recommended bonding agent mainly after abrasion with silica-coated Al_2O_3 particles. According to Silva et al,[36] a rough surface is prone to exhibit better wettability by increasing the surface area. However, in the current study, it was observed that the silane exhibited high capability of spreading over the zirconia surface, regardless of the surface roughness. This may explain the weak correlation between roughness and wettability observed in the present study. In the study of Yamaguchi et al[19] the same behavior is observed between roughness and surface free energy.

Although this study indicated that some groups seem to present more favorable micromechanical retention than the others, the investigation of the effect of these airborne-particle abrasion conditions on the adhesive bonding, as well as on the elemental composition, phase transformation and mechanical properties of the zirconia are necessary to confirm or not their efficacy.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions were drawn:

1. Although roughness and morphology of the zirconia surface varied according to the air-abrasion protocol, there was no close relation between them.
2. The roughness increase seemed to have followed the particle size enlargement.
3. Concerning micromechanical retention, the double airborne-particle abrasion, as recommended by the manufacturer, could be replaced by abrasion with only 120 μm Al_2O_3 particles.
4. The different air-abrasion protocols did not influence the wettability of the zirconia surface by the silane coupling agent.
5. No correlation between roughness and wettability was observed.

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TABLES

Table I. Materials evaluated

Material	Composition*	Manufacturer
50 μm Al_2O_3 particles	Al_2O_3>99%	Bio-Art Equip. Odontol. Ltda.
120 μm Al_2O_3 particles	Al_2O_3>99%	Bio-Art Equip. Odontol. Ltda.
250 μm Al_2O_3 particles	Al_2O_3>99%	Polidental Ind. Com. Ltda.
Rocatec Soft (30 μm)	aluminum oxide>97 wt% amorphous silica<3 wt%	3M ESPE AG
Rocatec Plus (110 μm)	aluminum oxide>95 wt% amorphous silica 1-5 wt%	3M ESPE AG
RelyX Ceramic Primer	MPS, ethanol, water	3M ESPE

MPS: 3-methacryloyloxypropyl trimethoxysilane

* informed by the manufacturers

Table II. Mean ± standard deviation of Ra (μm) and contact angle (º) values

	Ra	**Contact angle**
as-sintered (control)	0.35 ± 0.05[f]	1.66 ± 0.72*
50 μm Al_2O_3	0.52 ± 0.03[d]	1.60 ± 0.72*
120 μm Al_2O_3	0.80 ± 0.04[b]	1.50 ± 0.59*
250 μm Al_2O_3	1.13 ± 0.14[a]	1.56 ± 0.65*
Rocatec Soft (30 μm)	0.40 ± 0.03[e]	1.51 ± 0.49*
Rocatec Plus (110 μm)	0.69 ± 0.04[c]	1.45 ± 0.31*
120 μm Al_2O_3 + Rocatec Plus	0.79 ± 0.03[b]	1.32 ± 0.53*

[a,b,c,d,e,f] Different letters indicate significant differences ($P<.05$)

* No significant differences ($P>.05$)

LEGENDS FOR ILLUSTRATIONS

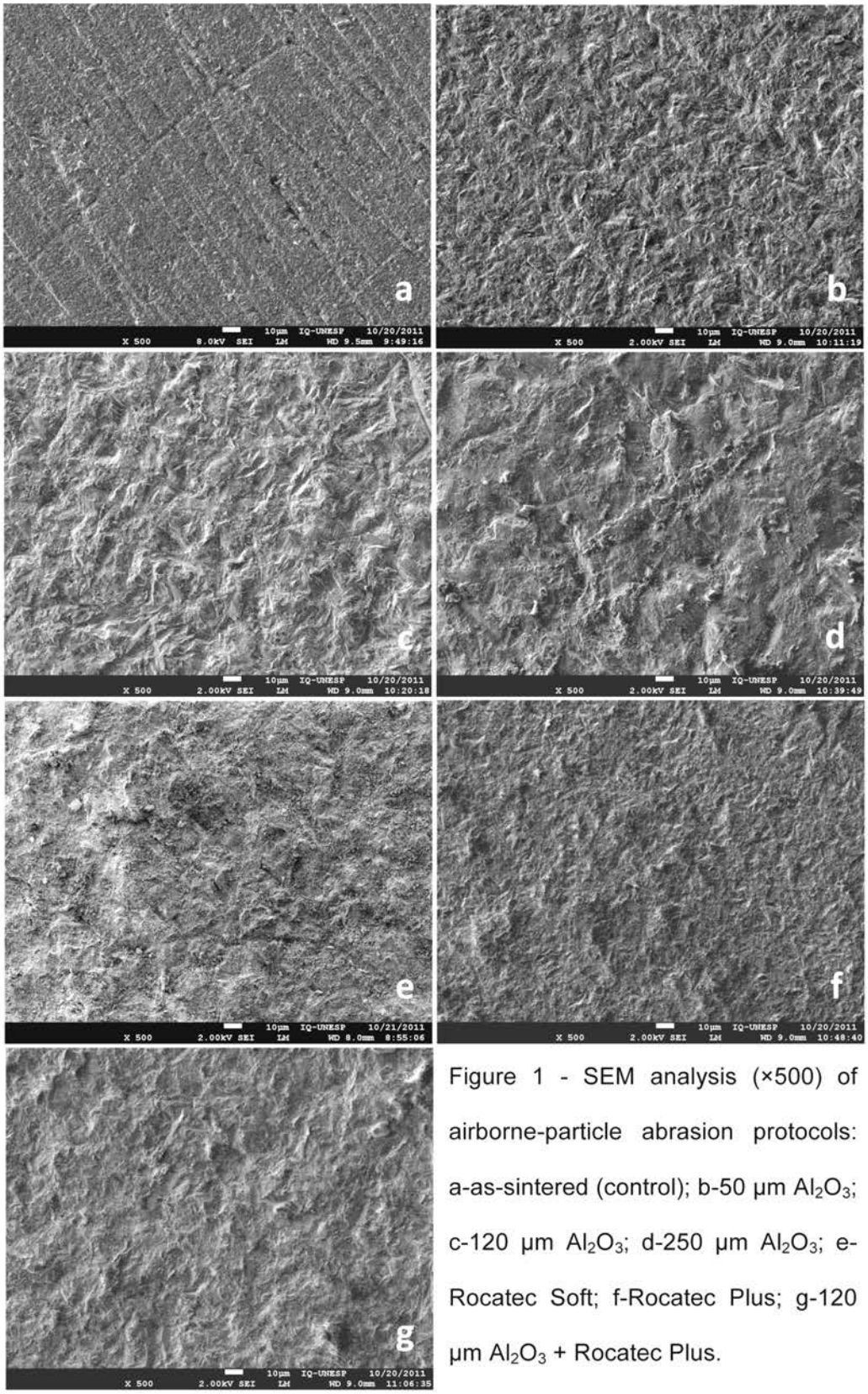

Figure 1 - SEM analysis (×500) of airborne-particle abrasion protocols: a-as-sintered (control); b-50 µm Al_2O_3; c-120 µm Al_2O_3; d-250 µm Al_2O_3; e-Rocatec Soft; f-Rocatec Plus; g-120 µm Al_2O_3 + Rocatec Plus.

3.2 CAPÍTULO 2

The effect of different air-abrasion moments on the Y-TZP ceramic surface characterization, flexural strength and shear bond strength to resin cement*

Air-abrasion moment effect on Y-TZP surface characterization, flexural and bond strength

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CLINICAL RELEVANCE STATEMENT

The air-abrasion performed before and after zirconia sintering can provide stronger bond strength at the zirconia/resin cement interface, as well as an increase in the short-term flexural strength.

SUMMARY

The purpose of this in vitro study was to evaluate the effect of air-abrasion moment on the yttria partially stabilized tetragonal zirconia polycrystal (Y-TZP) surface characterization (roughness, morphology and phase transformation), flexural strength (FS), and shear bond strength (SBS) to a resin cement. Y-TZP specimens were air-abraded with 50 μm Al_2O_3 particles after (AS); before (BS); or before and after zirconia sintering (BAS). For roughness (Ra), thirty block specimens (10 × 10 × 3.0 mm) (n=10) had their surfaces analyzed by a profilometer. Next, on the air-abraded surfaces of these specimens, composite resin discs (n=30) were bonded with RelyX ARC. The bonded specimens were stored for 24 h in distilled water at 37 ºC before shear testing. Failure mode was determined with a stereomicroscope (×20). The surface morphology (n=2) was evaluated by SEM (×500). For the 4-point flexural strength test (EMIC DL2000), thirty-nine bar-shaped specimens (20 × 4.0 × 1.2 mm) (n=13) were air-abraded according to the three moments proposed, and an additional group (non-abraded) was evaluated (n=13). The phase transformation (n=1) was analyzed by Rietveld refinement with X-ray diffraction data. Ra (μm) and SBS (MPa) data were analyzed by one-way ANOVA and Tukey's test (α=.05). Pearson correlation analysis was used to determine if there was a correlation between roughness and SBS. For FS (MPa) data, one-way ANOVA and Dunnett C test (α=.05) were

used. Air-abrasion moment was significant (*P*<.001) for Ra, SBS and FS data. The BS and AS groups presented the highest (1.3 μm) and the lowest (0.7 μm) Ra. The highest SBS (7.0 MPa) was exhibited by the BAS group, followed by the AS group (5.4 MPa) and, finally, by the BS group (2.6 MPa). All groups presented 100% adhesive failure. A weak correlation (r=-0.45, *P*<.05) was found between roughness and SBS. The surface morphology was influenced by the air-abrasion moment. The non-abraded (926.8 MPa) and BS (816.3 MPa) groups exhibited statistically similar FS values, but lower values than AS (1249.1 MPa) and BAS (1181.4 MPa) groups, with no significant difference between them. The non-abraded, AS, BS, and BAS groups exhibited, respectively, percentages of monoclinic phase of 0.0 wt%, 12.2 wt%, 0.0 wt%, and 8.6 wt%. The very rough surface provided by the air-abrasion before zirconia sintering impaired the bonding with the resin cement. The morphological patterns after the different air-abrasion moments were consistent with the surface roughness. Considering the short-term SBS and FS, the BAS group exhibited the best performance. Air-abrasion, regardless of its performance moment, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

Keywords: zirconia, air-abrasion moment, surface roughness, shear bond strength, morphology, phase transformation, flexural strength

INTRODUCTION

Yttria partially stabilized tetragonal zirconia polycrystal (Y-TZP) has been widely used to manufacture metal-free fixed partial dentures or implant-supported prostheses due to its optical properties,[1] biocompatibility,[2] low thermal conductivity,[3] chemical stability,[4] as well as its high fracture toughness and mechanical performance when compared to the other dental ceramics.[5]

In minimally retentive situations, resin cements are highly recommended[6] due to their improved mechanical properties when compared to zinc phosphate and glass ionomer cements,[7] and also because of the possible chemical interactions between zirconia surface and the resin cement components (adhesive cementation).[8,9] With regards to micromechanical retention, which is indispensable for improving the bonding between zirconia and resin cements,[10,11] although there are different methods to roughen zirconia surface, such as nano-structured alumina coating,[12] laser,[13,14] selective infiltration-etching (SIE),[15,16] hot etching solution,[16] among others, air-abrasion with alumina (Al_2O_3) particles is still an effective and one of the most applicable methods.[10,13,14,16]

Air-abrasion can be performed with Al_2O_3 particles of different sizes and is usually carried out after zirconia sintering and prior to cementation. However, since zirconia is a densely sintered material and consequently exhibits high hardness,[5] it is difficult to roughen its surface,[17] requiring higher air-pressure and/or coarser Al_2O_3 particles capable of promoting a desirable surface roughness. On the other hand, if this procedure is severe, it may create surface flaws, which can propagate into the bulk of the zirconia compromising its mechanical properties.[18,19] Another way to solve this question would be performing air-abrasion at a moment in which the zirconia does not exhibit such

high hardness, that is, before its sintering. This simple modification may allow the use of smaller particles to provide a surface whose roughness and morphology are favorable to the adhesive bonding at the zirconia/cement interface, without jeopardizing the mechanical strength of the zirconia. Monaco et al.[20] observed that, regardless of the particle size evaluated (30, 50 and 110 μm), the air-abrasion performed before zirconia sintering provided higher roughness in comparison with that performed after sintering. However, Monaco et al.[21] and Moon et al.[22] reported no significant differences in the shear bond strength between the groups abraded before and after zirconia sintering. Besides the increase in zirconia roughness reported by Monaco et al.,[20] another very important aspect observed by these authors, and also by Moon et al.,[22] was the decrease of the monoclinic phase when abrasion was performed before zirconia sintering.

Besides the few studies[20,22] that have investigated the effect of the air-abrasion moment (before sintering and after sintering) on zirconia roughness and adhesive bonding, there is no consensus with respect to the influence of the air-abrasion moment on roughness. Moreover, the association between the two air-abrasion moments, that is, before and after sintering, is another viable option to be investigated. In addition, it would be important not only evaluate the influence of different air-abrasion moments on phase transformation, but also on the mechanical strength of the zirconia.

Thus, the purpose of this in vitro study was to evaluate the effect of different air-abrasion moments (after, before, before and after zirconia sintering) on the Y-TZP ceramic surface characterization (roughness, morphology and phase transformation) and flexural strength (FS), and also its

efficacy on the shear bond strength (SBS) at the zirconia/resin cement interface. The null hypothesis was that different air-abrasion moments do not modify zirconia roughness nor its flexural strength and bond strength with a resin cement.

METHODS AND MATERIALS

Preparation of zirconia specimens

Thirty block specimens (12.5 × 12.5 × 3.5 mm) were prepared for roughness analysis and SBS test, while fifty-two bar-shaped specimens (25 × 5.0 × 1.5 mm) were prepared for 4-point flexural strength testing (ISO 6872[23]). The specimens were obtained by cutting pre-sintered zirconia frames (Lava; 3M ESPE AG, Seefeld, Germany) with a sawing machine (Isomet 1000; Buehler Ltd., Lake Bluff, IL, USA) using a diamond coated disc (Diamond Wafering Blade - Series 15LC Diamond nº 11-4276; Buehler Ltd., Lake Bluff, IL, USA) saw under water irrigation. The specimens were washed in tap water to remove the cutting debris and finished manually using a ceramic polisher (Exa Cerapol 0361HP; Edenta AG, Au, SG, Switzerland) in a low-speed handpiece.

The specimens were air-abraded with 50 μm Al_2O_3 particles (Bio-Art Equip. Odontol. Ltda., São Carlos, SP, Brazil) in 3 different moments: after zirconia sintering (AS); before zirconia sintering (BS); before and after zirconia sintering (BAS). For the 4-point flexural strength test, thirty-nine specimens were obtained according to the three air-abrasion moments proposed and an additional group (non-abraded) was included (n=13).

The sintering process was performed in a specific oven (Lava Furnace 200; Dekema Dental-Keramiköfen GmbH, Freilassing, Germany)

according to the manufacturer's instructions (heating rate=20 ºC/min: 0 ºC-1000 ºC; 10 ºC/min: 1000 ºC-1500 ºC; holding time=2 h and cooling rate=15 ºC/min: 1500 ºC-800 ºC; 20 ºC/min: 800 ºC-250 ºC - the oven was opened at 250 ºC). The dimensions of the specimens after sintering were 10 × 10 × 3.0 mm for roughness and SBS, and 20 × 4.0 × 1.2 mm for flexural strength. For the air-abrasion procedure, the specimens were mounted on a holder (developed for each specimen shape) at a 90-degree angle and a distance of 10 mm from the tip of the air-abrasion unit (Basic Classic; Renfert GmbH, Hilzingen, Germany).[24] The specimens were air-abraded for 20 s and 15 s at a pressure of 0.05 MPa and 0.28 MPa for abrasion before and after sintering, respectively. After sintering, all specimens were cleaned in 99% isopropanol using ultrasonic cleaner for 10 min, and left to dry in room temperature for 24 h. Both analyzes (surface roughness and SBS) were performed on the same specimens of each group.

Surface roughness measurements

The surface roughness of all specimens was determined after their sintering, using a profilometer (Surftest SJ-400; Mitutoyo Corporation, Kawasaki-shi, Japan) with a cut-off value (λ_c) of 0.8 mm.[14] A diamond stylus with a 5-µm tip radius at 0.5 mm/s and resolution of 0.01 mm examined a surface length of 2.4 mm. Three equidistant parallel measurements were made perpendicularly to the direction of the air-abrasion with a stylus at a 90-degree angle on different areas of the specimen. The average reading was designated as the Ra (µm) value of each specimen evaluated. A single calibrated operator (Intraclass Correlation Coefficient - ICC=0.89, Tabelas A1 e A2) recorded all measurements.

Bonding procedure and SBS test

Thirty composite resin discs (Z100; 3M ESPE, St. Paul, MN, USA) were produced using a custom-made metal split matrix (4.0 mm internal diameter and 2.0 mm thick) positioned between two glass slabs covered with transparent polyester films. The light curing (Radii-Cal light-curing unit; SDI Ltd., Bayswater, Australia) was performed for 40 s on the top surface and two diametrically opposed sides of the resin discs (total of 120 s) at a light intensity of 800 mW/cm^2. After the metal matrix was removed, the sides were light-cured taking care not to polymerize the bottom surface of the resin discs.

RelyX ARC resin cement (Bis-GMA, TEGDMA, silanated zirconia/silica filler – 3M ESPE, St. Paul, MN, USA) was proportioned by weight (0.010 g of each paste), mixed for 10 s, and the composite resin discs were immediately bonded to the air-abraded zirconia surfaces. Next, a load of 1000 g was applied on top of the composite resin disc for 5 min.[25] After excess removal, the cement was light-cured in two different positions (equidistant sides) for 40 s each.

The composite resin disc was inserted in a metal matrix (25 mm diameter) with a circular opening (4.2 mm diameter) with the zirconia block upwards. Polyvinyl chloride (PVC) tubes (20 mm in diameter and 20 mm high) were centrally positioned over the matrix and filled with polymethyl methacrylate (PMMA) autopolymerizing acrylic resin (Jet; Classico Odontological Goods Ltd., São Paulo, SP, Brazil), assembling the air-abraded zirconia surface to remain exactly at the same level of PMMA resin. All specimens were stored for 24 h in distilled water at 37 ºC.

Each specimen was mounted on a metal holder in a mechanical testing machine (model DL2000; EMIC Equipment and Systems Testing Ltd., São José dos Pinhais, PR, Brazil), and a uniaxial compressive force was applied at the cement/zirconia interface by means of a knife-edged blade running at a crosshead speed of 0.5 mm/min until failure. SBS values were recorded in MPa.

Failure analysis

Debonded specimens were examined under a stereomicroscope (model M80; Leica Microsystems Ltd., Heerbrugg, Switzerland) at ×20 magnification by a single calibrated observer and the failure mode was classified as: adhesive (complete zirconia surface was visible); cohesive within the cement layer or within the composite resin (almost all of the fracture surface was covered with cement or with composite resin) or mixed (a combination of adhesive and cohesive), according to the predominant mode of failure in each quadrant of the zirconia surface.[26]

Surface morphology analysis

For the surface morphology analysis, two additional specimens from each experimental group were mounted on metallic stubs, and analyzed under a field emission scanning electron microscope (model JSM-7500F; JEOL Ltd., Peabody, MA, USA), which operated at 500**×** magnification with an accelerating voltage of 2.0 kV.

Four-point flexural strength test

For the 4-point flexural strength test (ISO 6872 standard[23]), the specimens were positioned over two 0.8 mm-radius rounded bearers with a span distance of 16 mm. Two rounded loading pistons (0.8 mm-radius, distance of 8 mm) running at a crosshead speed of 1.0 mm/min applied a uniaxial compressive force to the non-abraded surface, while for the air-abraded groups the treated surface was submitted to the tensile load until failure. The test was performed at room temperature in a mechanical testing machine (model DL2000; EMIC Equipment and Systems Testing Ltd., São José dos Pinhais, PR, Brazil). The flexural strength values (MPa) were calculated according to the equation recommended by the ISO 6872 standard.

XRD analysis

The X-ray diffraction (XRD) analysis assessed the effect of the air-abrasion moments on the phase transformation of zirconia. Table 1 presents the scheme of XRD measurements according to the experimental groups.

The XRD data (n=1) were collected using a RIGAKU® RINT2000 rotating anode diffractometer (40 kV, 70 mA) with Cu kα radiation (λkα1=1.5405 Å, λkα2=1.5443 Å, Ikα1/Ikα2=0.5) monochromatized by a curved graphite crystal. An interval from 20º to 120° (2θ), with a step size of 0.02° (2θ), 4 s per step, divergence 0.5, and open receiving slits were the selected conditions for Rietveld refinement.[27] The Rietveld refinements were performed using the General Structure Analysis System (GSAS) program[28] suite with EXPGUI interface.[29] The peak profile function was modeled using a convolution of the Thompson-Cox-Hastings pseudo-Voigt function (pV-TCH),[30] using the asymmetry function

described by Finger et al.,[31] which accounts for the asymmetry resulting from axial divergence. The bi-dimensional model for crystallite size described by Larson and Von Dreele[28] was used to account for the anisotropy in the half width of the reflections, and the model described by Stephens[32] was used for an anisotropic strain analysis. The following parameters were refined: atomic coordinates, occupancies, unit cell, scale factor, sample displacement, atomic displacement and full width at half maximum (FWHM). The crystal structure parameter used as basis of the ICSD (Inorganic Crystal Structure Database) code was: 66781 (ZrO_2, tetragonal), 18190 (ZrO_2, monoclinic), and 53998 (ZrO_2, cubic).

Statistical analysis

The Shapiro-Wilk test indicated that the normality assumption for all data was satisfied, while the homogeneity by Levene test proved to be violated (*P*=.001) only for FS (MPa) data. Surface roughness (μm) and SBS (MPa) data were analyzed by one-way analysis of variance (ANOVA) followed by Tukey Honestly Significant Difference (HSD) post hoc test (α=.05) to determine differences among the means. In addition, to test for a possible correlation between roughness and SBS, a linear correlation ‘‘r’’ was calculated by Pearson correlation analysis. The analysis of FS (MPa) data was performed by one-way ANOVA and Dunnett C test (α=.05). Statistical analysis was performed using IBM SPSS Statistics (version 20; Statistical Package for Statistical Science Inc., Chicago, IL, USA).

RESULTS

According to the results of the one-way ANOVA, the air-abrasion moment was significant for surface roughness (*df*=2, F=70.1, *P*<.001), SBS (*df*=2, F=65.4, *P*<.001), and FS (*df*=3, F=12.0, *P*<.001).

Table 2 (Tabela A5) shows the mean Ra (μm) and SBS (MPa) values, standard deviations for each group, and statistical analysis results identified with Tukey HSD test. The BS group presented the highest Ra (μm), while the AS group yielded the lowest Ra value. The highest SBS value was exhibited by the BAS group, followed by the AS group and, finally, by the BS group. The failure mode observed was 100% adhesive in all groups. A weak correlation (r=-0.45, *P*<.05) was found between roughness and SBS.

The representative SEM images (Figure 1) indicated that the morphology of the zirconia surface was influenced by the air-abrasion moment.

Table 3 (Tabela A6) shows the FS (MPa) mean values, standard deviations and statistical results obtained by Dunnett C test. The non-abraded and BS groups exhibited statistically similar FS values, but lower values than AS and BAS groups, with no significant differences between them.

Table 4 lists the results of quantitative phase analysis and Figure 2 presents the representative diffraction patterns of the experimental groups according to each step performed to obtain them. Air-abrasion provided an increase in the monoclinic phase for the BS and BAS/abraded-1 groups, and a "decomposition" of t-ZrO_2 and c-ZrO_2 phases in others (t-ZrO_2 and m-ZrO_2) for AS and BAS/abraded-2 groups. The sintering process promoted the total incorporation of monoclinic phase into tetragonal and/or cubic phases.

DISCUSSION

The results of this study did not support acceptance of the null hypothesis, since air-abrasion moment influenced roughness, shear bond strength and flexural strength. The air-abrasion performed before zirconia sintering (BS group) provided the roughest surface (1.3 µm), followed by BAS (1.0 µm) and AS (0.7 µm) groups. Monaco et al.[20] also observed that the surface abraded with 50 µm Al_2O_3 particles before sintering exhibited higher roughness (2.33 µm) than that abraded after sintering (0.48 µm). These authors[20] suggest that the higher roughness showed by the BS group is due to the lower hardness of the zirconia in its green-stage. Although Moon et al.[22] did not find significant difference in roughness between the abrasion moments, these authors used 70 µm Al_2O_3 particles for the group abraded before sintering and 50 µm Al_2O_3 particles for that abraded after sintering, in order to compensate for the linear shrinkage of the zirconia during sintering. According to these authors,[22] the morphological analysis revealed that the group abraded before sintering presented a more blunted and melted-round surface while that abraded after sintering exhibited a coarse and needle-like rough topography. Conversely, Monaco et al.[20] observed that the group abraded with 50 µm Al_2O_3 particles before sintering exhibited a very rough surface, while a plastically deformed surface was observed in the group abraded after sintering. This finding corroborates the morphological pattern observed in the present study. The BAS group, which was not evaluated by Monaco et al.[20] and Moon et al.,[22] exhibited a surface texture similar to that presented by the BS group, but with more rounded edges probably resulting from the air-abrasion performed after sintering.

Considering that the effect of the abrasion moments has been poorly investigated, no additional information was found to further discuss our results.

Regarding the shear bond strength, the BS and BAS groups provided the lowest and the highest SBS values. Conversely, Moon et al.[22] reported that, for all cements evaluated, there was no significant difference in SBS between the groups abraded before and after sintering. This statistical similarity between these two abrasion moments was also observed by some authors.[21,24] In the current study, when the roughness values are compared with the SBS values, it can be observed that the highest roughness (1.3 µm) provided the lowest SBS (2.6 MPa). It is possible that the high roughness accompanied by the prominent edges observed by SEM prevented a suitable wettability of the abraded zirconia by the resin cement, and since a bonding agent was not used, this condition was intensified. On the other hand, the BAS group, which exhibited an intermediate roughness, promoted the highest SBS value (7.0 MPa), while the AS group exhibited the lowest roughness and a “flatter” morphology. Therefore, it seems that an intermediate roughness and morphology favored the adhesive bonding.

The weak correlation between roughness and bond strength observed in this study can be corroborated by the findings of Winkler and Moore.[33] These authors evaluated the correlation between these two properties varying the direction of the roughness measurements, that is, parallel or perpendicular to the scratches, and they concluded that when the reading was parallel, a correlation was observed. On the other hand, when the reading was perpendicular, as performed in the present study, the correlation was significantly lower. Also, in the study by Subaşı and Inan,[14] no significant correlation was

observed when the relationships between roughness and bond strength values were compared for each surface treatment and resin cement. Similarly, in the study of Oyagüe et al.,[25] although a correlation analysis was not performed, by observing the results of the zirconia roughness and microtensile bond strength, it seems that there is no correlation between these two variables.

Concerning flexural strength, the groups AS (1249.1 MPa) and BAS (1181.4 MPa) presented higher FS (with no significant difference between each other) than the non-abraded (926.8 MPa) and BS (816.3 MPa) groups (with no significant difference between each other). Although the BS group exhibited the lowest SBS value, it did not exhibit a decrease in the FS in comparison with the non-abraded group. It is possible that if a bonding agent (silane or adhesive monomers) had been applied after sintering, the wettability of the zirconia by the cement would be improved, resulting in higher SBS values.[11,13,34]

The XRD analysis performed for the BS group revealed a monoclinic phase content of 16.3 wt% after abrasion; however, after sintering, this percentage was zero. Moon et al.[22] observed the same behavior for the BS group, that is, a percentage of 16.9 wt% after abrasion, which dramatically decreased to almost zero. According to these authors, this behavior may be explained by the fact that air-abrasion itself induced tetragonal to monoclinic transformation, but a reverse transformation (monoclinic to tetragonal) occurred during the sintering process. Monaco et al.[20] reported that sintering zeroed the monoclinic phase that existed in the pre-sintering condition. Regarding the statistical FS superiority of the AS and BAS groups, it was probably due to the air-abrasion step. These two groups presented higher percentage values of monoclinic phase (AS=12.2 wt% and BAS=8.6 wt%) in comparison with the non-

abraded (0.0 wt%) and BS (0.0 wt%) groups. Using 50 μm Al_2O_3 particles, Monaco et al.[20] and Moon et al.[22] observed 10.0 wt% and 11.4 wt% of monoclinic phase for the group abraded after sintering. It is known that air-abrasion creates surface microcracks around which the grains exhibit a volumetric increase resulting from the tetragonal to monoclinic phase transformation. This outward expansion due to a plastic deformation of the surrounding zirconia provides compressive stresses, which counteract the crack propagation.[35] This process, known as transformation toughening, may increase the bulk strength of zirconia,[6,19,36-38] as indicated by this study.

Although this study did not evaluate the existence of a possible correlation between phase transformation (tetragonal to monoclinic) and flexural strength, it seems that there is some relation between them. Some studies[19,37] concluded that the increase in the mechanical performance of the zirconia seems to be related to the phase transformation (toughening mechanism), given that a higher amount of monoclinic ZrO_2 content resulted in higher flexural strength values. According to Souza et al.[38] the air-abrasion with alumina (50 and 110 μm) or silica-coated (30 and 110 μm) particles increased the biaxial flexural strength and the monoclinic content of zirconia when compared with the non-treated group. On the other hand, although some authors[39] had observed a higher percentage of monoclinic ZrO_2 after abrasion with 110 μm Al_2O_3 particles in comparison with the as-sintered zirconia, no significant difference in biaxial flexure strength was found between both treatments. This behavior is corroborated by the study of Borchers et al.,[40] who identified that different hydrothermal treatments provided a clearly detectable tetragonal to monoclinic phase transformation without any significant influence on the bulk strength of the

zirconia. According to these authors,[40] it is possible that the transformation zone did not penetrate deep enough into the zirconia to affect its mechanical strength. Therefore, a greater amount of monoclinic content, as a result of the tetragonal to monoclinic transformation, is not always related to the material's greater mechanical strength.

Besides the lack of studies that compared the air-abrasion moment routinely performed (AS group) with that performed before sintering (BS group), the novelty of the current research is that the combination of both air-abrasion moments was tested, and yielded the best results with regards to the shear bond strength and flexural strength. However, a concern that should be taken into account is the behavior of the non-desirable microcracks in the three air-abrasion moments. In the BS group, microcracks are created by air-abrasion, resulting in phase transformation (tetragonal to monoclinic), which contains their propagation. After sintering, an inverse phase transformation occurred (monoclinic to tetragonal),[22] releasing the compressive stresses,[20,37] which is not so damaging given that the zirconia has a sintering shrinkage of about 20%-25%, which could promote a partial or total sealing of the cracks.[41] This fact may explain the similar behavior concerning the FS of the BS group in comparison with the non-abraded one. Although the BS group exhibited lower FS than the AS and BAS groups, in the long term, it may behave more favorably under cyclic load and moisture. On the other hand, in the AS (the air-abrasion moment routinely performed) and BAS groups, the microcracks created by the air-abrasion after sintering were probably contained by the compressive stresses resulting from the phase transformation (tetragonal to monoclinic).[19,36-38] This fact may explain the higher FS of these groups in comparison with the BS one.

However, we wonder whether the condition of the AS and BAS groups is maintained for a sufficiently long period of time under the adverse effects of the oral environment.

Therefore, it is essential to investigate how these conditions resulting from the air-abrasion moments evaluated behave under long-term moisture environment, which favors the propagation of microcracks due to the low temperature degradation phenomenon, and under cyclic loading, in order to simulate the adverse conditions of the oral cavity.

CONCLUSIONS

Within the limitations of this in vitro study, the following conclusions may be drawn:

1. The very rough surface provided by the air-abrasion before zirconia sintering impaired the bonding with the resin cement.
2. The morphological patterns after the different air-abrasion moments were consistent with the surface roughness.
3. Considering the short-term SBS and FS, the BAS group exhibited the best performance.
4. Air-abrasion, regardless of its performance moment, provides tetragonal to monoclinic transformation, while sintering tends to zero the monoclinic phase content.

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LEGENDS

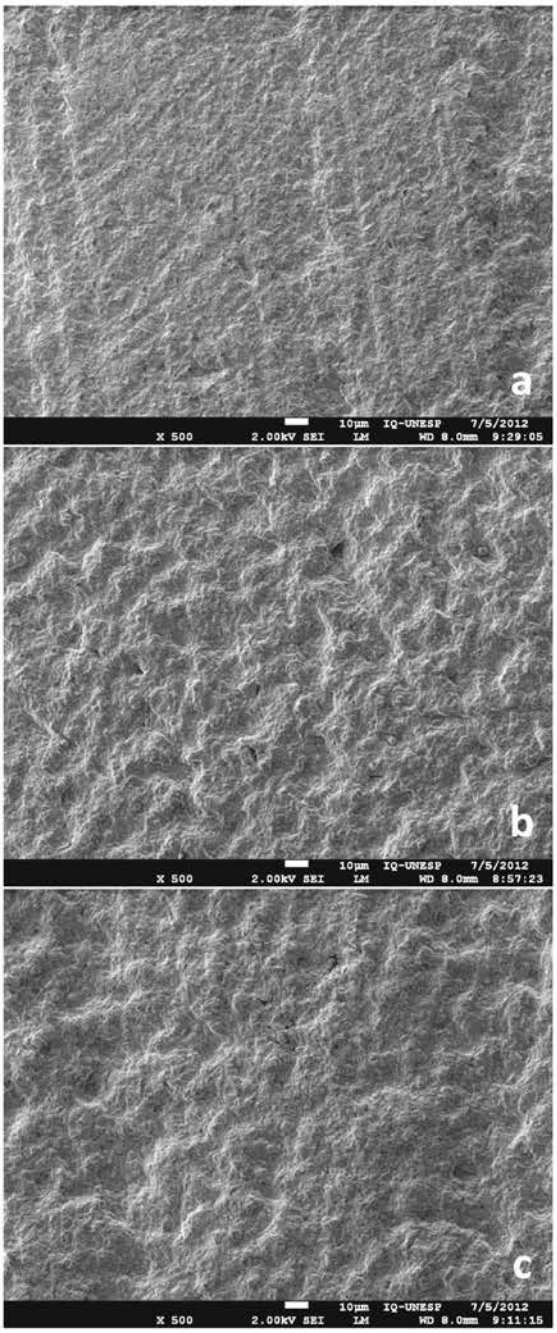

Figure 1. SEM analysis (×500) of the air-abrasion moments: a - after zirconia sintering (AS); b - before zirconia sintering (BS); c - before and after zirconia sintering (BAS).

TABLES

Table 1. Scheme of XRD measurements

non-abraded	**AS**	**BS**	**BAS**
non-abraded non-sintered	non-abraded sintered	air-abraded non-sintered	air-abraded-1 non-sintered
1st measurement	1st measurement	1st measurement	1st measurement
sintering process	air-abrasion procedure	sintering process	sintering process
2nd measurement	2nd measurement	2nd measurement	2nd measurement
-	-	-	air-abrasion procedure (air-abraded-2)
-	-	-	3rd measurement

Table 2. Mean ± standard deviation of Ra (µm) and SBS (MPa) values

	Ra	**SBS**
AS	0.7 ± 0.1[c]	5.4 ± 0.6[b]
BS	1.3 ± 0.1[a]	2.6 ± 0. 9[c]
BAS	1.0 ± 0.1[b]	7.0 ± 1.1[a]

[a,b,c] Different letters indicate significant differences in columns (*P*<.05)

Table 3. Mean ± standard deviation of FS (MPa) values

	FS
non-abraded	926.8 ± 95.4[b]
AS	1249.1 ± 303.9[a]
BS	816.3 ± 112.4[b]
BAS	1181.4 ± 262.7[a]

[a,b] Different letters indicate significant differences (*P*<.05)

Table 4. Phase content (wt%) of the experimental groups

Groups	**Conditions**	**Phases (wt%)**			
		t-ZrO_2	**m-ZrO_2**	**t-ZrO_2**	**c-ZrO_2**
non-abraded	non-sintered	85.5(1)	14.4(1)	-	-
	sintered	89.2(1)	-	-	10.7(1)
AS	as-sintered	89.2(1)	-	-	10.7(1)
	abraded	59.2(5)	12.2(6)	28.6(1)	-
BS	abraded	83.7(7)	16.3(3)	-	-
	sintered	74.6(2)	-	-	25.4(7)
BAS	abraded-1	83.5(7)	16.5(3)	-	-
	sintered	73.2(3)	-	-	26.8(8)
	abraded-2	51.0(4)	8.6(2)	40.3(5)	-

t - tetragonal, m - monoclinic, c - cubic

Considerações Finais

4 CONSIDERAÇÕES FINAIS

Para a realização do jateamento, estão disponíveis partículas de óxido de alumínio (Al_2O_3) convencionais e revestidas por sílica de diferentes tamanhos (25 µm a 250 µm). Já está suficientemente comprovado que o jateamento aumenta a rugosidade da superfície da zircônia. Geralmente, a análise da rugosidade em função de diferentes tamanhos de partículas indica que partículas maiores tendem a promover maior rugosidade. Quanto à morfologia, a textura da superfície jateada também parece tornar-se mais acentuada/marcada à medida em que se aumenta o tamanho da partícula. De uma forma geral, o padrão morfológico é condizente com a rugosidade, ou seja, o aumento da rugosidade é acompanhado por um aumento na textura de superfície. Entretanto, pode haver também um aumento da rugosidade e paralelamente, uma deformação plástica ou “achatamento” revelados pelas imagens morfológicas.

O jateamento, além de limpar e aumentar a área de superfície disponível para a união do cimento à zircônia, promove alterações físico-químicas, aumentando a energia livre de superfície, o que pode favorecer o molhamento. No presente estudo, diferentes partículas empregadas no jateamento não promoveram diferença significativa no molhamento. Na literatura, a investigação da influência do tamanho da partícula no molhamento é escassa. No entanto, sabe-se que as partículas de Al_2O_3 revestidas por sílica podem apresentar maior energia livre de superfície. Em um estudo recente realizado pelo nosso grupo de pesquisa, observou-se que tais partículas promoveram maior molhamento quando o silano foi utilizado. Essas duas propriedades

dependem da afinidade entre o substrato e o líquido. Para um mesmo substrato, líquidos diferentes podem resultar em comportamentos diferentes, sendo também o inverso verdadeiro. Com relação à influência da partícula no molhamento, esta propriedade parece depender da interação entre líquido e substrato e, também, aumentar com o aumento da rugosidade, porém, até certo limite, ou seja, rugosidade muito elevada não implica necessariamente em elevado molhamento.

Com relação ao momento do jateamento, no presente estudo, observou-se que a falta ou o excesso de rugosidade prejudicam a resistência de união da interface zircônia/cimento, indicando fraca correlação entre rugosidade e resistência. O ideal seria uma rugosidade intermediária, em torno de 1,0 µm, para permitir um escoamento mais adequado do cimento nas microrretenções da superfície da zircônia.

Rotineiramente, o jateamento é realizado na zircônia sinterizada. Este procedimento cria microtrincas na superfície do material, as quais são contidas pelas forças compressivas resultantes da transformação de fase, podendo até resultar em aumento imediato da resistência mecânica da zircônia. Entretanto, sob efeito das condições orais adversas, como carga cíclica e umidade, as microtrincas podem se propagar em direção ao corpo da zircônia, causando redução da sua resistência. Tal fato nos levou a investigar o efeito da realização do jateamento antes da sinterização da zircônia, uma vez que não há estudos relacionados. O jateamento realizado neste momento, apesar de ter apresentado o menor valor de resistência adesiva e flexural, parece ter preservado as características da zircônia, no tocante à resistência mecânica e transformação de fase e, além disto, deixou a zircônia em uma condição mais

favorável (ausência de fase monoclínica e possível contenção/selamento das microtrincas) diante das condições adversas supracitadas.

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Apêndice

APÊNDICE

Tabela A1 - Valores de rugosidade (µm) e ângulo de contato (º), obtidos em duas leituras diferentes, para a Análise do Coeficiente de Correlação Intraclasse (CCI). Faculdade de Odontologia de Araraquara, 2014

	Rugosidade		Ângulo de Contato*	
	1ª. leitura	**2ª. leitura**	**1ª. leitura**	**2ª. leitura**
1	0,70	0,68	69,20	65,50
2	0,74	0,72	61,70	61,10
3	0,79	0,78	63,10	62,60
4	0,83	0,79	65,50	65,30
5	0,64	0,61	67,00	68,50
6	0,77	0,80	69,80	66,80
7	0,70	0,71	68,70	70,00
8	0,77	0,75	65,30	68,10
9	0,73	0,72	68,40	68,00
10	0,73	0,76	68,00	69,80
11	0,67	0,69	67,00	66,90
12	0,68	0,70	62,70	66,00
13	0,71	0,72	70,00	68,60
14	0,74	0,77	68,50	66,00
15	0,69	0,67	65,10	62,00

* a mensuração do ângulo de contato para a calibração do operador foi realizada com água destilada

Tabela A2 - Classificação dos valores de Coeficiente de Correlação Intraclasse de acordo com a Proposta de Fermanian*. Faculdade de Odontologia de Araraquara, 2014

P	**Concordância**
<0,31	Nula
0,31–0,51	Medíocre
0,51–0,71	Moderada
0,71–0,91	Boa
0,91–1,00	Excelente

* Fermanian J. Measuring agreement between 2 observers: a quantitative case. Rev Epidemiol Sante Publique. 1984;32:408-13.

Tabela A3 - Valores originais, médias, desvios-padrão (DP) e coeficientes de variação (CV) da rugosidade (µm) após os diferentes protocolos de jateamento. Faculdade de Odontologia de Araraquara, 2014

	controle	Al_2O_3 50 µm	Al_2O_3 120 µm	Al_2O_3 250 µm	Rocatec Soft	Rocatec Plus	Al_2O_3 120 µm + Rocatec Plus
1	0,29	0,55	0,83	1,34	0,43	0,72	0,80
2	0,37	0,59	0,78	1,24	0,34	0,68	0,84
3	0,26	0,56	0,85	1,25	0,39	0,68	0,80
4	0,31	0,52	0,87	1,16	0,41	0,73	0,79
5	0,44	0,56	0,80	1,16	0,37	0,68	0,77
6	0,35	0,54	0,82	1,28	0,39	0,69	0,77
7	0,36	0,52	0,86	1,29	0,44	0,75	0,77
8	0,27	0,52	0,81	1,26	0,42	0,72	0,85
9	0,35	0,52	0,81	1,23	0,43	0,73	0,83
10	0,35	0,53	0,83	1,11	0,37	0,74	0,81
11	0,38	0,53	0,75	1,01	0,41	0,68	0,79
12	0,36	0,52	0,72	1,04	0,38	0,65	0,78
13	0,36	0,54	0,83	1,18	0,37	0,66	0,79
14	0,33	0,51	0,81	1,08	0,36	0,65	0,76
15	0,28	0,45	0,76	1,01	0,39	0,65	0,74
16	0,42	0,47	0,77	1,19	0,46	0,69	0,82
17	0,37	0,52	0,76	0,86	0,37	0,70	0,76
18	0,41	0,47	0,74	0,85	0,4	0,62	0,76
19	0,33	0,49	0,77	1,04	0,47	0,71	0,8
20	0,36	0,49	0,76	0,92	0,4	0,66	0,79
Média	**0,35**	**0,52**	**0,80**	**1,13**	**0,40**	**0,69**	**0,79**
DP	**0,05**	**0,03**	**0,04**	**0,14**	**0,03**	**0,04**	**0,03**
CV (%)	**13,84**	**6,51**	**5,28**	**12,84**	**8,51**	**5,10**	**3,64**

Tabela A4 - Valores originais, médias, desvios-padrão (DP) e coeficientes de variação (CV) do ângulo de contato (º) após os diferentes protocolos de jateamento. Faculdade de Odontologia de Araraquara, 2014

	controle	Al_2O_3 50 µm	Al_2O_3 120 µm	Al_2O_3 250 µm	Rocatec Soft	Rocatec Plus	Al_2O_3 120 µm + Rocatec Plus
1	0,40	3,10	1,10	1,20	1,80	1,20	1,30
2	0,70	2,80	1,60	1,60	1,00	1,60	0,90
3	0,80	2,70	1,20	1,70	1,90	1,60	1,10
4	1,50	1,90	0,90	1,20	1,40	1,50	1,80
5	1,50	1,10	1,80	0,80	1,90	1,80	0,70
6	1,60	1,00	1,30	0,70	0,70	1,70	1,10
7	1,50	2,90	1,60	0,70	1,80	1,80	0,60
8	1,90	1,80	0,40	1,30	1,70	1,00	1,50
9	1,50	1,20	0,80	1,10	0,70	1,20	1,30
10	1,00	1,60	1,30	1,20	1,60	1,70	0,60
11	1,50	1,30	1,60	1,60	2,00	2,10	2,10
12	2,80	1,30	1,50	1,60	1,90	1,10	1,00
13	0,70	1,00	2,20	2,90	2,30	1,40	1,60
14	2,10	1,00	1,90	0,90	1,30	1,00	2,50
15	2,90	1,10	0,90	2,60	1,50	1,80	1,40
16	2,00	0,90	2,40	2,40	1,40	1,20	2,10
17	1,90	1,20	2,60	2,50	1,10	1,40	1,80
18	2,90	1,60	0,80	1,80	1,60	1,10	1,00
19	2,10	1,50	1,70	2,00	2,00	1,50	0,80
20	1,90	0,90	2,30	1,30	0,50	1,30	1,20
Média	**1,66**	**1,60**	**1,50**	**1,56**	**1,51**	**1,45**	**1,32**
DP	**0,72**	**0,72**	**0,59**	**0,65**	**0,49**	**0,31**	**0,53**
CV (%)	**43,24**	**45,00**	**39,75**	**41,65**	**32,73**	**21,40**	**40,28**

Tabela A5 - Valores originais, médias, desvios-padrão (DP) e coeficientes de variação (CV) da rugosidade – Ra (µm) e resistência de união ao cisalhamento – RC (MPa) após os diferentes momentos do jateamento. Faculdade de Odontologia de Araraquara, 2014

	Pós-sinterização		**Pré-sinterização**		**Pré- e Pós-sinterização**	
	Ra	**RC**	**Ra**	**RC**	**Ra**	**RC**
1	0,58	5,34	1,18	3,73	0,85	8,57
2	0,59	5,47	1,22	2,68	0,89	5,58
3	0,60	4,53	1,21	3,22	0,95	6,41
4	0,63	6,14	1,23	2,32	0,96	5,35
5	0,67	4,60	1,23	1,65	0,98	6,60
6	0,68	5,88	1,27	1,35	1,05	7,01
7	0,70	5,36	1,28	3,5	1,03	7,03
8	0,80	5,01	1,33	1,7	1,12	8,23
9	0,80	5,59	1,34	3,65	1,12	8,12
10	0,93	6,12	1,48	2,22	1,27	7,07
Média	**0,70**	**5,40**	**1,28**	**2,60**	**1,02**	**7,00**
DP	**0,11**	**0,57**	**0,09**	**0,89**	**0,13**	**1,08**
CV (%)	**16,27**	**10,49**	**6,88**	**34,08**	**12,25**	**15,43**

Tabela A6 - Valores originais, médias, desvios-padrão (DP) e coeficientes de variação (CV) da resistência à flexão 4 pontos (MPa) após os diferentes momentos do jateamento. Faculdade de Odontologia de Araraquara, 2014

	Controle (não jateado)	**Pós-sinterização**	**Pré-sinterização**	**Pré- e Pós-sinterização**
1	908,15	1750,96	818,77	1205,44
2	893,74	783,13	853,36	1516,51
3	868,54	1069,00	990,41	754,59
4	1007,94	782,04	801,16	1298,53
5	1075,72	944,78	873,93	1257,20
6	963,40	1350,41	721,48	732,37
7	1002,45	1262,29	677,78	868,87
8	821,15	1404,41	994,58	1565,21
9	706,28	1216,31	823,64	1366,00
10	923,04	1102,13	612,99	1342,81
11	1002,73	1581,37	784,96	1111,50
12	974,49	1554,15	746,42	1197,70
13	900,93	1437,13	912,30	1141,28
Média	**926,81**	**1249,08**	**816,29**	**1181,39**
DP	**95,38**	**303,89**	**112,43**	**262,68**
CV (%)	**10,29**	**24,33**	**13,77**	**22,24**

Araraquara, 20 de março de 2014.

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