UNESP - Universidade Estadual Paulista

"Júlio de Mesquita Filho"

Faculdade de Odontologia de Araraquara

THIAGO SOARES PORTO

Análise comparativa de diferentes materiais utilizados para confecção de restaurações em CAD/CAM

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Tese apresentada ao Programa de Pós-Graduação em Ciências Odontológicas, Área de Dentística Restauradora, da Faculdade de Odontologia de Araraquara, da Universidade Estadual Paulista “Júlio de Mesquita Filho” – UNESP, para obtenção do Título de Doutor em Ciências Odontológicas.

Orientador: Prof. Dr. Edson Alves de Campos

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Salmo 91(90)

O justo confia em Deus

Eu que habito ao amparo do Altíssimo,
e vivo a sombra do Onipotente,
digo a Javé: "Meu refúgio, minha Fortaleza,
meu Deus, eu confio em ti!"

Ele me livrará do laço do caçador,
e da peste destruidora.
Ele me cobrirá com suas penas,
e debaixo de suas asas eu me refugiarei.
O braço dele é meu escudo e minha armadura.
Eu não temerei o terror da noite,
nem a fleche que voa de dia,
nem a epidemia que caminha nas trevas,
nem a peste que devasta ao meio-dia.
Caiam mil ao meu lado
e dez mil à minha direita,
a mim nada atingirá.

Basta que eu olhe com meus próprios olhos,
para ver o salário dos injustos,
porque eu fiz de Javé o meu refúgio
e tomou o Altíssimo como meu defensor.
A desgraça jamais me atingirá,
e praga nenhuma vai chegar à minha minha tenda.
pois ele ordenou aos meus anjos
que me guardem em meus caminhos.
Eles me levarão nas mãos,
para que meu pé não tropece numa pedra.
Eu caminharei sobre cobras e víboras,
e pisarei leões e dragões.

"Eu o livrarei, porque a mim se apegou.
Eu o protegerei, pois conhece o meu nome.
Ele me invocará, e eu responderei.
Na angústia estarei com ele.
Eu o livrarei e glorificarei.
Vou saciá-lo de longos dias
e lhe farei ver a minha salvação".

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“Existem, durante a nossa vida, sempre dois caminhos a seguir:
Aquele que todo mundo segue, e
aquele que a nossa imaginação nos leva a seguir.
O primeiro pode ser o mais seguro, o mais confiável,
o menos crítico... Mas, você será apenas mais um a caminhar.
O segundo com certeza vai ser o mais difícil, mais solitário,
o que você terá maiores
críticas... Mas também, o mais criativo, o mais original possível.
Não importa o que você seja, ou o que deseja na vida, a ousadia em ser diferente reflete na sua personalidade, no seu caráter, naquilo que você é.
E é assim que as pessoas lembrarão de você um dia.”

Ayrton Senna da Silva

Porto TS. Análise comparative de diferentes materiais utilizados para confecção de restaurações em CAD/CAM [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2016.

RESUMO

O objetivo deste estudo é investigar e comparar, por meio de três diferentes protocolos de pesquisa, o comportamento de diferentes materiais cerâmicos utilizados para fabricação de restaurações em CAD/CAM. No Estudo 1, o objetivo foi determinar quais dos materiais possui a melhor usinabilidade, ou seja, a facilidade de corte. Os blocos para CAD/CAM selecionados foram: IPS e.max CAD, IPS Empress CAD, Vitablocs Mark II e LAVA Ultimate, estes cortados em uma máquina de corte (Isomet 4000, Buehler, Lake Bluff, IL, USA) e levados ao microdurômetro. Através das medidas da identação e trincas geradas pela leitura, foi determinado qual dos materiais utilizados em restaurações de CAD/CAM possui a melhor usinabilidade por meio do cálculo do índice de fragilidade. O LAVA Ultimate apresentou a melhor usinabilidade seguido pelo IPS Empress CAD. O IPS e.max CAD e o Vitablocs Mark II não apresentaram diferenças estatisticamente significantes. Para os estudos 2 e 3 os materiais selecionados foram IPS e.max CAD, Vitablocs Mark II, LAVA Ultimate e Enamic. No Estudo 2, o objetivo foi determinar qual dos materiais possui maior resistência à fratura após serem submetidos a diversas condições de termociclagem. Os blocos de cerâmicas de CAD/CAM foram cortados em várias fases até atingirem as dimensões finais de 14mm x 3mm x 2,5mm, no formato de barra, foram confeccionados um total de 144 corpos de prova. A resistência à fratura foi determinada por meio do teste de três pontos com um entalhe realizado nos corpos de prova. Posteriormente as fraturas apresentadas foram analisadas em microscópio eletrônico de varredura (MEV) e classificadas quanto as fraturas ocorridas. O melhor desempenho foi obtido pelo material IPS

e.max CAD, nestes um aumento estatisticamente significante foi observado após 120.000 termo ciclos. Os materiais LAVA Ultimate e Enamic apresentaram uma redução na resistência à fratura após a termociclagem.

No Estudo 3, o objetivo foi determinar a melhor performance entre os materiais do ponto de vista da resistência flexural e módulo de elasticidade. Os corpos de prova possuíam as mesmas medidas do estudo 2, porém sem o entalhe na parte inferior. Igualmente também submetidos as mesmas condições de termociclagem, do estudo 2. Como resultado sem levar em consideração a termociclagem o material com a maior resistência flexural foi o IPS e.max CAD, seguido em ordem decrescente pelo Vita Enamic, Lava Ultimate e Vitablocs Mark II. Por outro lado na avalição do modulo de elasticidade o material com o menor valor foi Lava Ultimate, devido a sua composição baseada em compósito. Composição esta que levou o material a sofrer influência da termociclagem, fazendo com que sua resistência flexural diminuisse de maneira estatisticamente significante após 60.000 e 120.000 ciclos. Porém entre os outro materiais avaliados não houveram alterações em suas propriedades mecânicas a serem consideradas. Importante ainda salientar que nos estudos 2 e 3 as fotomicrografias realizadas em MEV evidenciaram fraturas de características frágil para as cerâmicas e o Vita Enamic, com exceção do Lava Ultimate que apresentou fraturas com característica dúcteis.

Palavras–chave: materiais dentários, cerâmica, propriedades físicas

Porto TS. Comparative analysis of different CAD/CAM materials used to indirect restorations [Tese de Doutorado]. Araraquara: Faculdade de Odontologia da UNESP; 2016.

ABSTRACT

The aim of this study was to investigate and compare, through three different research protocols, the behavior of different ceramic materials used to manufacture CAD/CAM restorations. In Study 1, the objective was to determine which material has better machinability, i.e., ease of cutting. The blocks for CAD/CAM selected were: IPS e.max CAD IPS Empress CAD, VITABLOCS Mark II and LAVA Ultimate, these were cut on a cutting machine and taken to microhardness. From the measurements of the indentation and crack generated by the readings was determined which of the materials used for CAD/CAM restorations has the better machinability by calculating the Brittleness index. The LAVA Ultimate showed the best machinability followed by IPS Empress CAD. The IPS e.max CAD and VITABLOCS Mark II showed no statistically significant differences. For studies 2 and 3 the selected materials were IPS e.max CAD, VITABLOCS Mark II, LAVA Ultimate and Enamic. In Study 2, the goal was to determine which of the materials have greater fracture toughness after being subjected to various conditions of thermal cycling. Ceramic CAD/CAM blocks were cut in several stages until got the final dimensions of 14mm x 3mm x 2.5mm on bar formats, in total 144 specimens were made. The fracture toughness was determined by the three-point bending test with a notch made in the specimens. Later fractures were analyzed in scanning electron microscope and classified how the fractures occurred. The best performance was shown by the IPS e.max CAD material, where a statistically significant increase was observed after 120,000 cycles term. The LAVA Ultimate and Enamic materials showed reduction in their fracture toughness after thermal cycling.

In Study 3, the objective was to determine the best performance among the materials on flexural strength and flexural modulus. The specimens had the same measures of the study 2, but without the notch on the bottom. Also underwent the same conditions of thermal cycling the study 2. As a result without taking into account the thermal cycling the material with higher flexural strength was the IPS e.max CAD, followed in descending order by Vita Enamic, Lava Ultimate and Vitablocs Mark II. In the other hand, taking account elasticity modulus the material with the lowest value was Lava Ultimate due to its composite composition. This composition took the material to be influenced by thermal cycling, making its flexural strength impaire statistically significant way after 60,000 and 120,000 cycles. However among the other materials tested there were no changes in their mechanical properties to be considered. Also important to note that in studies 2 e 3 photomicrographs made by SEM showed brittle fractures for ceramics and Vita Enamic, except for Lava Ultimate which showed fractures with ductile characteristics.

Keywords: dental materials, ceramic, physical properties,

Lista de Ilustrações

ARTIGO 1

ARTIGO 2

ARTIGO 3

Figure 1 – Vitablocs Mark II – categorized as felsdpathic porcelain, at the 120x magnification show an appearance known as fish scale in the direction of the fracture, identified by the white arrow. A brittle fracture occurred by cleavage is apparent over the surface among the two different magnifications, also conchoidal fracture is visible as result of breakage surface that appears like a wavelet.

Figure 2 – IPS e.max CAD – classified as lithium disilicate glass ceramic, show peaks and valleys on the 120x magnification due to action of needle-like crystals randomly oriented. The crystals cause the deflection of cracks propagation providing a substantial increase in the flexural strength. A brittle fracture is clear on the ceramic surface and flaws are also apparent. Porous region characterize the center of the circle shown at 800x magnification.

Figure 3 – Lava Ultimate – the resin nano ceramic material show ductile fracture characteristics, at the 120x magnification can be identified at the right side of figure by the undulation. Closer (800x magnification) some small undulations and flaws are also visible characterizing the material with plastic behavior.

Figure 4 – Enamic – a polymer-infiltrated-ceramic-network besides the polymer composition, the fracture characteristics point out a material with ceramic behavior. At the 120x magnification the fish scale feature are clearly identified as is for feldspathic ceramic. Complementing at 800x magnification the ceramic network is visible and no plastic deformations were found. On the surface of both magnification is apparent some porous region and also handling damage.

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APÊNDICE

SUMÁRIO

1 INTRODUÇÃO

A busca pela excelência nas reabilitações protéticas é crescente e muitos são os parâmetros a serem estudados nesse desafio. Podemos citar entre os fatores que mais podem influenciar um bom resultado reabilitador, as propriedades físicas e mecânicas dos materiais, a adaptação marginal das restaurações e sua linha de cimentação (Cho et al.,[11] 2011; Conrad et al.,[13] 2007; Kramer, Frankenberger,[26] 2005; Pallesen, van Dijken,[34] 2000). Durante muito tempo o ouro foi o material eleito em restaurações indiretas devido às suas características de adaptação aos preparos, contato proximal, polimento e manuseio laboratorial (Stappert et al.,[50] 2008; Wagner et al.,[55] 2003). Por razões estéticas, as restaurações metálicas, de um modo geral, têm sido amplamente substituídas pelos sistemas cerâmicos livres de metal (Blatz,[6] 2002; Giannetopoulus et al.,[20] 2010; Manhart et al.,[32] 2004; Pallense, van Dijken,[34] 2000; Wagner et al.,[55] 2003). De acordo com evidências na literatura, pode-se dizer que os resultados clínicos de restaurações cerâmicas, após 10 anos em função, são muito similares ao ouro e não seria possível afirmar qual é a melhor restauração (Wagner et al.,[55] 2003).

Apesar dos trabalhos protéticos metalo-cerâmicos ainda serem os mais utilizados para reabilitações unitárias ou múltiplas em região posterior, as coroas de infraestrutura metálica possuem suas desvantagens inerentes ao material, como por exemplo: a) oxidação metálica, que pode levar ao manchamento da margem gengival; b) retração gengival devido aos íons metálicos; c) exposição do metal na região cervical; d) falta de opalescência nas coroas devido à transmissão de luz desfavorável; entre outras (Bernardes et al.,[4] 2012; Giannetopoulus et al.,[20] 2010; Lorenzoni et al.,[29] 2010). Essas desvantagens começaram a ser questionadas de maneira mais abrangente devido ao aumento da exigência estética, além da busca de um material restaurador com melhores propriedades mecânicas, físicas e biológicas, o que impulsionou o desenvolvimento das restaurações em cerâmica pura (El-Mowafy, Brochu,[17] 2002).

Embora as coroas unitárias posteriores confeccionadas em cerâmica pura mostrem um tempo de sobrevida significativamente menor do que as metalo-cerâmicas (Pjetursson et al.,[35] 2007), investigações clínicas realizadas por alguns autores (Davidowitz, Kotick,[15] 2011; Lorenzoni et al.,[29] 2010; Pallense, van Dijken,[34] 2000; Pjetursson et al.,[35] 2007; Rosenblum, Schulman,[41] 1997) mostram que as coroas de cerâmicas puras têm excelentes propriedades estéticas, ótima estabilidade de cor e estão constantemente melhorando sua resistência frente aos esforços exigidos durante a mastigação. Embasados por artigos científicos (Blatz,[6] 2002; Manhart et al.,[32] 2004) podemos dizer que existe uma tendência crescente em substituir restaurações com bases metálicas por cerâmicas puras e ainda afirmar que coroas unitárias feitas por diferentes cerâmicas (disilicato de lítio, leucita, alumina) têm sido utilizadas por mais de 30 anos com excelente durabilidade e estão se tornando referências em grandes reabilitações (Blatz,[6] 2002; Manhart et al.,[32] 2004).

Quanto as cerâmicas porém, ainda é necessário mais estudos e aprofundamento de pesquisas para melhor avaliar as propriedades mecânicas que são essenciais para o entendimento de suas aplicações. A caracterização micro estrutural e a determinação destas propriedades são os primeiros passos para entender de uma maneira geral o comportamento dos materiais utilizados em Odontologia Restauradora, como por exemplo a utilização da microscopia eletrônica de varredura (MEV), que é uma ferramenta utilizada para demonstrar características importantes em diversas situações da topografia dos materiais, além de propiciar a visualização de parâmetros micro estruturais, a saber; tamanho e forma das partículas cerâmicas (Borba et al.,[9] 2011; Della Bona et al.,[16] 2014; Rodrigues et al.,[40] 2008; Sinmazisik, Ovecoglu,[46] 2006). A associação do MEV com espectroscopia de energia dispersiva, permite a melhora da qualidade na quantidade de informação química das diferentes fases do material. Algumas importantes características relacionadas ao comportamento desses materiais quando em função são essenciais na pesquisa, densidade, modulo de Young, Poisson ratio são alguns exemplos, que em conjunto com analise de elemento finito criam um vasto campo de estudo (Corazza et al.,[14] 2013; Geng et al.,[19] 2001).

O domínio das técnicas de aglutinação e sinterização em forno específico, sendo cada camada estratificada de acordo com a necessidade estética, permitiu obter nas reabilitações um perfeito mimetismo com as propriedades ópticas dos tecidos remanescentes, que além de serem atrativas, têm biocompatibilidade com os tecidos bucais (Bernardes et al.,[4] 2012). No entanto, o complexo processo de fabricação das cerâmicas laboratoriais ainda é feito prioritariamente com a utilização de técnicas seculares que não evoluíram nos últimos tempos. Essas técnicas envolvem materiais com diferentes propriedades e características (Bernardes et al.,[4] 2012), cuja manipulação consome muito tempo do ceramista pelo fato de ter muitos passos e variáveis, exigindo habilidade específica do mesmo ao coordenar simultaneamente olhos e mãos (Christensen et al.,[12] 2006). Além desses aspectos podemos mencionar também fatores que favorecem a dificuldade em determinar a longevidade das próteses, como: formato e espessura do material de infraestrutura, a aplicação da cerâmica, métodos laboratoriais de fabricação e módulo de elasticidade das próprias cerâmicas (Goodacre et al.,[21] 2003; Heintze et al.,[29] 2008).

Sabemos que para o sucesso da reabilitação é importante a precisão de adaptação das coroas ao substrato dentário. A tecnologia CAD/CAM (computer aided design/computer aided manufacturing) tem como grande vantagem a confecção computadorizada de restaurações minimizando as pequenas distorções causadas pelos métodos convencionais, além de eliminar várias etapas laboratoriais que podem ser fatores complicadores em sua confecção (Goodacre et al.,[21] 2003; Heintze et al.,[29] 2008). Assim sendo, o constante desenvolvimento das restaurações livres de metal e a tecnologia uniram as cerâmicas e o

CAD/CAM há anos, e a evolução destes materiais cerâmicos, assim como do sistema digital têm sido crescente (Pallense, van Dijken,[34] 2000).

Podemos afirmar que a tecnologia CAD/CAM foi desenvolvida para resolver três grandes desafios: 1) Proporcionar à restauração resistência adequada às forças mastigatórias as quais é submetida, quando comparada com as restaurações de cerâmicas convencionais; 2) Criar restaurações com uma aparência mais natural; e 3) Fazer com que as reabilitações se tornem mais fáceis, rápidas e precisas. Em alguns casos, a tecnologia proporciona a finalização de reabilitações no mesmo dia em que se inicia o trabalho (Batalha-Silva et al.,[2] 2013; Davidowitz, Kotick,[15] 2011; Pallesen, van Dijken,[34] 2000).

A tecnologia CAD/CAM promove inúmeras possibilidades de interação entre profissional clínico e laboratório de prótese, facilitando o trabalho do dentista e do protético. O dentista pode moldar as coroas de maneira convencional utilizando as siliconas para impressão, ou então utilizar uma câmera intra-oral no ato de fazer a impressão digital e enviar ao laboratório, ou mesmo confeccionar o trabalho ele mesmo se possuir uma fresadora de consultório. Da mesma maneira, o protético pode dar sequência à confecção das coroas quando recebe a impressão convencional, confeccionando o modelo de gesso e escaneando o modelo para obtenção da imagem digital com um escâner de laboratório, ou quando recebe do dentista a imagem de escaneamento digital, seguindo através do computador e fazendo todo o trabalho de *design* através do *software* a partir das imagens recebidas (Boccaccini,[8] 1996).

A utilização de CAD/CAM permite a confecção de preparos cavitários mais conservadores, o que torna a restauração mais resistente (Giannetopoulus et al.,[20] 2010; Magne, Knezevic,[31] 2009; Saridag et al.,[43] 2013). Do mesmo modo, materiais como resinas compostas de laboratório podem ser mais facilmente confeccionados em máquinas CAD/CAM, com propriedades mais desejáveis devido às condições ideais às quais são realizados (Magne, Knezevic,[31] 2009; Saridag et al.,[43] 2013), pela simples confecção digital da restauração final. No entanto estudos mostram comportamentos inesperados como uma resistência maior à fadiga dessas resinas quando comparadas às cerâmicas, muito provavelmente devido ao módulo de elasticidade ser similar ao da dentina, o que nos possibilita, além de mais opções restauradoras, uma mudança na escolha de qual material utilizar em determinadas situações (Kelly,[24] 1999; Magne, Knezevic,[30] 2009; Magne et al.,[31] 2010).

Muitas pesquisas tem estudado a performance clinica de coroas feitas por materiais resinosos (Behr et al.,[3] 2003; Rammelsberg et al.,[39] 2005; Vannorbeek et al.,[54] 2010). Alguns desses estudos mostram que coroas de compósitos, utilizando a técnica de incremento, tem um performance aceitável com uma média de 96% após 3 anos conforme relata Rammelsberg et al.[39] (2005). Outro estudo realizado por Vannorbeek et al.[54] (2010), reporta após 3 anos uma média de 87.9% de coroas feitas por materiais resinosos ainda em função, essas coroas foram realizadas através da tecnologia CAD/CAM. Os estudos mostram como razão principal de falha a fratura, e sugerem que a resistência das coroas de materiais resinosos é um dos principais fatores para obter um resultado clinico adequado.

Mesmo assim é importante checar a literatura perante as principais propriedades mecânicas. A resistência à fratura é considerada uma das propriedades principais no comportamento clinico das cerâmicas dentais (Harada et al.,[22] 2015). A resistência à fratura caracteriza a capacidade do material à resistir a propagação de trincas, e consequentemente à falha prematura, com especial relevância à fratura de materiais frágeis (Scherrer et al.,[44] 1998; Yilmaz et al.,[56] 2007). Para materiais cerâmicos, o método recomendado para determinar a resistência à fratura é o teste através da pré-trinca realizada no corpo de prova. O corpo de prova deve ser confeccionado no formato de uma barra, que mostra-se pela literatura de fácil confecção, repetição e precisão.

Assim diante do crescente aumento de técnicas e materiais protéticos é possível visualizar uma lacuna nas possíveis interações entre propriedades mecânicas, e a usinabilidade dos materiais, que pode ser calculada de maneira quantitativa através de seu índice de fragilidade, e as suas qualidades de adaptação marginal, após testes mecânicos, como: ciclagem termomecânica e resistência à compressão. Heintze et al.[23] em 2008 concluíram que para avaliar coroas cerâmicas em metal free é indispensável a utilização da carga dinâmica para conclusões válidas de performance clínica. Do mesmo modo, Saridag et al.[43] em 2013 também avaliaram a resistência à fratura de restaurações cerâmicas puras, em dois tipos de preparos diferentes (inlay e onlay), além do controle. Eles concluem (Saridag et al.,[43] 2013) que para o IPS e.max Press, a resistência à fratura é menor nos preparos onlay; já no grupo cerâmica de zircônia (ICE Zirkon) o tipo de preparo não influenciou a resistência.

Seguindo a mesma linha de pensamento podemos citar outros trabalhos (Heintze et al.,[23] 2008; Magne e Knezevic,[30] 2009; Magne et al.,[31] 2010; Stappert et al.,[50] 2008) em que é investigado a influência da fadiga após movimentos mastigatórios utilizando também a ciclagem térmica para o envelhecimento da amostra. As discrepâncias na integridade marginal visualizadas pelos autores em réplicas de resina epóxica mostram que essas desadaptações marginais no sentido vestíbulo-lingual foram significativamente mais altas do que as mesio-distais em todos os estágios. Entre os materiais por eles utilizados as restaurações de ouro evidenciaram superioridade marginal seguida pelo cerômero. As restaurações de cerâmica tinham margens aceitáveis, porém a fabricação por CAD/CAM comprometeu levemente a integridade marginal, segundo os autores.

Uma avaliação clinica realizada por Calamia et al.[10] (1989) onde foram avaliados 115 facetas laminadas de porcelana condicionadas e cimentadas por um único pequisador clinico, trabalho este desenvolvido na Universidade de Nova Iorque entre os anos de 1982 e 1985. As restaurações foram divididas em dois grupos, onde o primeiro grupo consistia de 43 restaurações cimentadas sem qualquer tipo de preparo dental, e o segundo grupo foram instaladas 72 restaurações após o preparo de um chanfro. Os critérios avaliados foram, a descoloração marginal na margem cavo-superficial, integridade marginal e presença de lesões cariosas. O exame clinico revelou também o número de falhas de cimentação e fraturas da porcelana. Uma avaliação periodontal mostrou mínimas respostas negativas ao tratamento e o baixo índice de falhas no estudo, associado a nenhuma incidência de cárie indicaram que as restaurações tiveram um resultado satisfatório.

Segundo Qualtrough et al.[37] (1991), não há consenso a respeito da margem de preparo para restaurações inlay. O estudo realizado por esses autores comparou preparos realizados com acabamento variando entre margens com bisel e sem bisel. Vinte e quatro preparos classe II MOD foram definidas como preparo padrão em pré-molares extraídos. Doze foram realizados utilizando o acabamento com bisel e 12 sem bisel. Restaurações em porcelana foram fabricadas e instaladas com material resinoso de polimerização dual. As restaurações finalizadas foram estocadas em água, termocicladas, e analisadas utilizando-se um microscópio eletrônico de varredura (MEV). A qualidade da interface esmalte/material resinoso foi confirmada sendo consideravelmente melhor que a interface porcelana/material resinoso. A adaptação do material resinoso ao esmalte foi de igual qualidade para ambos, preparos biselados e não biselados.

Na pesquisa realizada por Mörmann e Krejci,[33] (1992), oito mesio-ocluso-disto *inlays* adesivas, foram fabricadas a partir de blocos cerâmicos utilizando o sistema "*computer-aided design/computer-machining method"* e foram reavaliadas após 5 anos em função. As restaurações foram avaliadas de acordo com o critério do Serviço Público de Saúde dos Estados Unidos. Os resultados indicaram excelente performance clinica, com oito classificações Alfa para desgaste, caries recorrentes, e estabilidade de cor; e cinco Alfa e três Bravos para descoloração marginal e integridade marginal. Após a leitura no microscópio eletrônico de varredura, 81% das interfaces dente-cimento e 84,1% das interfaces cimento-inlay foram classificadas com interface sem defeitos na margem oclusal. Axialmente, 73,6% das interfaces dente-cimento, e 87% das interfaces cimento-inlay também foram classificadas sem defeitos. Apesar disso a investigação no longo prazo revelou boa performance clinica, com melhoras na infraestrutura cerâmica e suas propriedades, além do mais o design da margem cavo-superficial, e o material resinoso de cimentação foram recentemente introduzidos para otimizar a qualidade.

Diferentes métodos para determinar a resistência à fratura de cerâmicas no modo I tem sido descritas na literatura. A precisão e reprodutibilidade entre examinadores depende do teste realizado, e dos parâmetros utilizados nas condições dos corpos de prova. Scherrer et al.[44] (1998), propuseram o estudo de comparar a resistência à fratura por métodos diferentes de identação, assim como um novo método estabelecido por fratura mecânica. Aplicaram os seguintes métodos; (1) fratura por identação (IF), (2) força de identação (IS) e (3) teste do corpo de prova em forma de barra com pré-trinca. Os materiais testados foram cerâmicas com baixo ponto de fusão (Duceram LFC) e cerâmica feldspática (IPS classic). Para ambos os materiais, o coeficiente de resistência à fratura teve uma variação entre 10% e 14% para IF e 7% e10% para IS. A força de identação demonstrou uma dependência da força aplicada para a porcelana IPS, qual não foi vista para o método de fratura por identação. O teste mecânico mostrou resultados consistentes com coeficientes de variação entre 1% e 3%. A resistência à fratura para a porcelana IPS estava em acordo com o teste IS realizado. Apesar disso, foram observadas trincas na pré-fratura realizada de todos os corpos de prova do Duceram mostrando talvez valores subestimados. Como conclusão os autores relatam que os testes realizados obtiveram sucesso e são reprodutíveis, além de mostrarem semelhança maior de 90%. Porém não se comportam exatamente iguais e deve ser levado em consideração um entendimento das características cerâmicas assim como as limitações para definir o a técnica mais adequada.

Com a melhora nas propriedades mecânicas dos materiais livres de metal, há algum tempo pesquisas investigam a utilização desses materiais na região posterior. Conforme relata Rammelsberg et al.[38] (2000), a resistência à fratura de materiais livres de metal não deve ser considerado um fator limitante para a utilização em coroas posteriores. Como objetivo no trabalho fizeram a avaliação axial das paredes do preparo, desgaste oclusal realizado e técnica de cimentação na resistência de coroas realizadas em *Artglass*. Utilizaram setenta e dois terceiros molares extraídos, que receberam preparos padronizados. A parede axial foi realizada de duas maneiras; com desgaste de 1mm formando um ombro e com desgaste de 0,5mm formando um chanfro, e o desgaste oclusal igualmente em duas medidas 0,5mm e 1,3mm. As coroas de Artglass realizadas foram cimentadas com três cimentos diferentes: fosfato de zinco, ionômero de vidro e um cimento resinoso combinado com adesivo dental. Após a termociclagem de 10.000 ciclos entre 5^oC e 55^oC, as coroas artificiais foram verticalmente levadas até a fratura. Nos resultados a mínima resistência a fratura encontrada foi com a força de 500N, porém 9 de 24 coroas cimentadas com fosfato de zinco, soltaram após a termociclagem. A cimentação adesiva resultou em significativa melhora na resistência à fratura comparada com o ionômero de vidro e cimento de fosfato de zinco. Aumento da espessura oclusal, resultou em melhor estabilidade, e o desgaste da parede axial não resultou em melhoras. Concluíram que o desgaste axial de 0,5mm associado com uma redução oclusal adequada e cimentação adesiva garante excelente estabilidade posterior para coroas livres de metal.

Tanoue et al.[52] (2006) conduziu um trabalho avaliando a influência da força centrifuga no carregamento de partículas em compósitos utilizando a polimerização com luz combinada com a centrifugação. Para acessar a distribuição das partículas resultantes da centrifugação dos corpos de prova, duas resinas de baixa viscosidade (Palfique Estelite LV e Revolution Formula 2) foram selecionadas e colocadas em tubos de teste, centrifugadas e consequentemente polimerizadas. Após cada corpo de prova ser cortado em quatro discos de 2mm de espessura, as partículas inorgânicas e a dureza Knoop foram determinadas. Os resultados encontrados sugeriram que o carregamento de partículas em compósito, pode ser melhorado através da aplicação da força centrifuga, se o monômero e as partículas forem proporcionalmente corretas.

A caracterização da microestrutura e determinação de algumas propriedades mecânicas foi proposta e investigada por Della Bona et al.[16] (2014), como forma de conhecer melhor e entender as características de materiais para CAD/CAM conhecidos como cerâmicas infiltradas por polímeros. Corpos de prova foram fabricados com o objetivo de analisa-los quantitativamente e qualitativamente quanto a sua microestrutura, além de determinar sua resistência à fratura, densidade, *Poisson ratio* e modulo de *Young*. A resistência à fratura foi determinada através de espécimenes confeccionados no formato de barras, com uma pré-trinca realizada, e sob o teste de carregamento foram fraturados. A densidade foi calculada utilizando o principio de Arquimedes, e o *Poisson ratio* e o modulo de *Young* foram medidos através de ondas ultrassônicas combinados com um osciloscópio pulsado. Os resultados encontrados por esses autores mostram a microestrutura formado por cerâmica e uma base de polímero penetrada. As médias e desvio padrão das propriedades avaliadas foram: resistência à fratura 1,09±0,05Mpa $m^{1/2}$; densidade 2,09±0,01g/cm^3; Poisson ratio 0,23±0,002; e modulo de Young 37,95±0,34GPa. Concluíram que as cerâmicas infiltrados por polímeros tem propriedades mecânicas entre porcelanas e materiais a base de compósito, refletindo seus componentes microestruturais.

Com o objetivo de estudar se o processo de fabricação de coroas cerâmicas feitas pelo sistema CAD/CAM ou pelo processo manual de aplicação afetam a resistência à fratura, Harada et al.[22] (2015) selecionaram os seguintes matérias: Lava Ultimate, Estenia C&B, e o IPS e.max Press. A partir desses materiais quatro tipos diferentes de coroas foram preconizadas. Coroas CAD/CAM feitas com o Lava Ultimate; técnica manual de construção com camada única com Estenia C&B; técnica manual de construção com várias camadas com Estenia C&B; e coroas cerâmicas infiltradas com IPS e.max Press. Cada tipo de coroa foi cimentada em preparos e a fratura à resistência foi testada. As coroas Estenia multicamadas mostraram significativamente menor resistência à fratura quando comparadas com as coroas de Lava e IPS e.max Press, apesar disso não houve diferença na resistência a flexão e resistência à fratura quando os materiais foram comparados através de corpos de prova com formato de barras. Tomografia micro-computadorizada e análise fractográfica mostraram que a diminuição da resistência provavelmente resulta de falhas internas nas coroas realizadas através das multicamadas. Não houve diferença significativa na resistência à fratura entre Lava, Estenia C&B camada única, e IPS e.max Press. Ambos os tipos de coroa de material resinoso mostraram fraturas maiores que 2000N, oque é maior que a força de mordida na região posterior. As coroas CAD/CAM realizaram coroas, sem os defeitos internos, e poderiam ser aplicadas a região de molar com suficiente resistência à fratura.

Em uma pesquisa recente realizada por Kelvin Khng et al.[25] (2016), foi identificado que a precisão de coroas feitas pelo sistema CAD/CAM não foi completamente investigada e ainda existem dúvidas quanto a avaliação da integridade marginal. Com o propósito de um estudo *in vitro* eles estudaram a avaliação da parte interna, relacionada a integridade marginal de coroas fabricadas através do sistema CAD/CAM e as compararam com coroas convencionais fabricadas em polimetilmetacrilato (PMMA). Um pré-molar inferior esquerdo foi preparado com características para receber uma coroa de cerâmica e escaneado para a fabricação de 60 preparos em resina. Metade dos preparos foram escaneados para fabricação de 15 coroas Telio CAD-CEREC e 15 coroas Paradigm MZ 100-E4D-E4D. Trinta coroas provisórias sendo 15 Caulk e 15 Jet foram confeccionadas nos preparos remanescentes. Todas as coroas foram cimentadas com Tempgrip sob uma força de 17.8N, termocicladas por 1000 ciclos, então colocadas em ácido fucsinico, e por ultimo posicionadas em resina epóxica antes da secção. A discrepância marginal foi medida através de microscópio óptico, e através da porcentagem de penetração do ácido no contorno sob a coroa. Como resultados a média vertical de discrepância encontrada nas coroas convencionais foi maior que as fabricadas em CAD/CAM, enquanto nenhuma diferença foi encontrado no componente horizontal. Uma menor porém significante discrepância marginal foi encontrada nas coroas fabricadas pelo sistema CAD/CAM quando comparadas com as coroas convencionais; diferença que não foi observada no componente horizontal. A porcentagem de penetração do ácido foi correlacionada com as discrepâncias verticais e horizontais na superfícies axiais das coroas.

Uma recente pesquisa conduzida por Song et al.[49] (2016) calculando a fragilidade e usinabilidade de cerâmicas estéticas e compostas por disilicato de lítio, evidenciou a importância não somente dos procedimentos de fabricação através do sistema CAD/CAM como a também o processo de finalização e ajuste. A usinabilidade neste estudo foi feita simulando a cavidade oral, utilizando uma peça de mão e pontas diamantadas. As forças geradas através da velocidade e energia necessária para corte foram calculadas por um mecanismo de alta velocidade com um sensor de força. As superfícies usinadas do disilicato de lítio foram visualizadas através de um perfilômetro óptico confocal e microscópio eletrônico de varredura. As superfícies e morfologia das pontas diamantadas utilizadas foram também avaliadas. Mínimo desgaste das ferramentas porém diversos processos de delaminação foram visualizados. As forças e energia de usinagem dependeram significativamente das condições de fresagem e foram significativamente maiores que outras cerâmicas vítreas. As velocidades de usinagem caíram drasticamente comparadas com outras cerâmicas vítreas. Os índices de usinabilidade associados com dureza, modulo de Young e resistência à fratura colocam o disilicato de lítio no ranking das cerâmicas vítreas mais difíceis de serem usinadas.

É importante salientarmos, ainda, trabalhos de outros autores sobre possíveis fatores que poderiam influenciar a resistência e a adaptação dos materiais cerâmicos. Esses autores (Taira, Yamaki,[51] 1994; Tsitrou et al.,[53] 2007) mediram a usinabilidade de formas diferentes, através de corte das cerâmicas e também através do cálculo do seu índice de fragilidade por meio da dureza Vickers. Segundo os autores (Tsitrou et al.,[53] 2007), o grau de desadaptação de uma restauração usinada pode ser estimado calculando o fator de desadaptação marginal (Marginal Chipping Factor). Os resultados mostraram que o cerômero Paradigm MZ100 (3M/ESPE) mostrou os menores valores de índice de fragilidade (BI) e fator de desadaptação marginal (CF), enquanto o IPS e.max CAD mostrou os maiores valores.

No estudo que realizamos, foram utilizados os seguintes materiais: LAVA Ultimate (3M ESPE); IPS e.max CAD (Ivoclar-Vivadent); IPS Empress CAD (Ivoclar-Vivadent); Vitablocs Mark II (Vita Zahnfabrik-Vident); Enamic (Vita Zahnfabrik-Vident). Ao analisar a ficha técnica do LAVA Ultimate, vemos que em sua composição ele possui 85% de partículas ultrafinas de cerâmicas sílica-zircônia, que reforça a matriz polimérica, a qual é formada por BisGMA e TEGDMA. Comparando com a composição química do IPS e.max CAD percebemos a grande diferença entre os materiais, pois este é constituído em sua maior parte de cerâmica vítrea de di-silicato de lítio (SiO_2); possui também óxido de lítio (Li_2O), óxido de potássio (K_2O), óxido de magnésio (MgO), óxido de alumínio (Al_2O_3), pentóxido de fósforo (P_2O_5), entre outros óxidos. Ainda pensando na relação entre os dois materiais e analisando suas características frente a esforços vemos que, o IPS e.max possui uma resistência à flexão de 360 MPa e um módulo de elasticidade de 95 GPa, enquanto o LAVA Ultimate tem em torno de 150 MPa de resistência à flexão e um módulo de elasticidade próximo de 12 GPa. Assim, correlacionando o trabalho dos autores Tsitrou[53] et al. realizado em 2007 com a composição química dos materiais por eles observados, pode-se levantar uma hipótese de que os blocos para CAD/CAM de resina composta, assim classificados pelo fabricante, podem ter uma performance clínica superior às cerâmicas. Porém, seria necessário avaliá-los quanto a sua resistência e durabilidade diante das condições encontradas no meio bucal ou em situações que simulem estas condições.

Cerâmicas e materiais resinosos são as duas classes principais de materiais restauradores. Materiais resinosos são normalmente compostos por um polímero com matriz orgânica e reforçados com partículas inorgânicas (Ferracane,[18] 1995). A quantidade de partículas pode estar diretamente relacionada com o módulo de elasticidade e a dureza desses materiais. O desenvolvimento de uma tecnologia de inclusão de partículas inorgânicas teve consideráveis avanços nas propriedades mecânicas dos materiais resinosos. Uma importante atenção deve ser dada no momento de seleção das partículas inorgânicas pois, está pode ter uma influência direta nas características ópticas, e segundo trabalhos publicados na literatura partículas de sílica compõe as exigências necessárias. Por outro lado, as alterações dimensionais resultantes da polimerização são determinadas pelos monômeros da matriz polimérica, e os mais comuns são Bis-GMA, UDMA, UTMA, e Bis-EMA (Ferracane,[18] 1995). Ao analisarmos a performance clinica das restaurações diretas de resina composta comparadas com trabalhos cerâmicos indiretos ainda são consideradas inferiores no que tange a adaptação marginal, propriedades ópticas e forma anatômica.

É nessa lacuna que esta investigação se insere, considerando que analisamos o índice de fragilidade de materiais estéticos e sua relação com a resistência e adaptação.

Sabemos, ainda, que as cerâmicas vítreas possuem diversas composições e algumas delas são menos resistentes, podendo comportar-se com similaridade aos cerômeros, o que limita sua utilização em coroas e restaurações unitárias na região anterior. Porém, as cerâmicas de zircônia são extremamente resistentes e podem ser utilizadas para reabilitações múltiplas de um modo geral (Lorenzoni et al.,[29] 2010). No entanto, o ideal seria desenvolver um material com a resistência de uma cerâmica e a resiliência de uma resina, mas para isso ainda são necessárias inúmeras pesquisas. No desenvolvimento dos compósitos e cerâmicas para reabilitação, a busca por resistência e resiliência é constante. Recentemente a Vita Zahnfabrik lançou um material buscando essas características, utilizando uma nova tecnologia e uma nova classificação de substrato. Conhecido como Enamic, uma cerâmica infiltrada por polímero, este material não é um compósito e tampouco é cerâmica pura segundo informações do fabricante; é uma mistura de ambos, o que aumenta ainda mais as possibilidades de eleição de materiais nas inúmeras situações de trabalho.

Temos um campo de pesquisa extremamente vasto e interessante quando consideramos o grande número de cerâmicas e compósitos existentes e suas diversas composições, proporcionando a esses materiais diferenças em sua dureza, resistência e durabilidade. Alguns parâmetros de resistência e adaptação já estão estabelecidos; porém, ainda não está claro na literatura qual material se comporta melhor frente aos esforços mastigatórios e principalmente se sua resistência ou técnica de confecção tem alguma relação com suas características de adaptação. Há perguntas ainda não respondidas como: será que adaptação marginal pode ser influenciada pela dureza do material ou pela técnica de confecção? E ainda sendo o material mais resistente, pode-se afirmar que as deformações e/ou fraturas de esforços não poderiam ser minimizadas por materiais que tenham um módulo de elasticidade mais alto?

2 PROPOSIÇÃO

2.1 Objetivo Geral

Com base na literatura apresentada, o objetivo geral do presente trabalho foi avaliar a usinabilidade dos materiais restauradores estéticos por meio de sua dureza, e ainda avaliar a resistência à fratura, resistência flexural e modulo de flexão destes quando submetidos à termociclagem. Com posterior indentificação do comportamento da fratura por análise fractográfica. Assim serão avaliados:

2.2 Objetivos específicos

2.2.1 Índice de fragilidade e usinabilidade de materiais restauradores estéticos através da dureza Vickers;

2.2.2 Resistência à fratura após termociclagem e tipos de fraturas por análise fractográfica;

2.2.3 Resistência flexural e módulo de elasticidade após termociclagem e tipos de fraturas por análise fractográfica.

3 PUBLICAÇÕES

3.1 Publicação 1

Title: *Brittleness index assessment and its relationship with the Vickers hardness: influence on the machinability of ceramic materials**

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Keywords: ceramics, dental porcelain, CAD/CAM, chipping, brittle ceramics, brittleness index, glass ceramic

* Artigo de acordo com as normas do periódico Dental Materials

Abstract

Objective: The aim of this study was to evaluate the brittleness index and the Vickers hardness of different materials CAD / CAM and the interaction between their properties.

Methods: Fifty-two specimens made of four different materials were performed: Vitablocks Mark II (Vita Zahnfabrik) as a control group, IPS e.max CAD (Ivoclar-Vivadent), IPS Empress CAD (Ivoclar-Vivadent), and Lava Ultimate (3M ESPE). Slices were made out from the blocks with the following dimensions 14 x 10 x 3 mm (L x W x H), using a cutting machine with a diamond disk and then regularized through a polishing device. The brittleness index was calculated by measuring the crack along the apex of the indentation in the same direction of the diagonal 1. The Vickers Hardness was calculated by measuring the diagonals 1 and 2.

Results: For the brittleness index materials IPS e.max CAD obtained the highest values (2:53 ± 0:17), while the Lava Ultimate had the lowest values (0.91 ± 0.03). The results related to Vickers hardness also had the IPS e.max CAD with the highest average (697.71 ± 16:50) and again the resin nano ceramic Lava Ultimate with the lowest average (102.81 ± 6:50). The calculation of Pearson correlation showed a positive relationship between the two variables.

Significance: The brittleness index also regarded as the machinability of a material, was evident within this study as positively dependent on the Vickers hardness, what leads us to conclude that the hardness of the ceramic is related to its milling capacity.

1. Introduction

The pursuit of excellence in aesthetic and prosthetic rehabilitation is growing and there are many parameters to be studied in this challenge. Despite all factors that can influence rehabilitation; mechanical materials properties, restorations marginal fit and the cementing process [1-4] are important keys to achieve the sucess. For a long time the gold was the best material for indirect restorations due to adaptation characteristics, polishing and laboratory handling [5-6]. However, according to literature evidence, after 10 years in function clinical findings of ceramic restorations are very similar to gold [6].

Porcelain fused to metal (PFM) are still the most widely used for single or multiple restorations in posterior region, however, metal infrastructure have material inherent disadvantages such as: a) metallic oxidation, related to staining gingival margin; b) gingival recession due to metal ions; c) metal exposure in the cervical region; and d) lack of opalescence crowns due to unfavorable light transmission [7-9]. These disadvantages have started to be questioned more extensively due to increased aesthetic demand, which spurred the development of pure ceramic restorations [10].

Rehabilitation success is dependent from precision crowns to dental substrate. The computer-aided-design and computer-aided-manufacturer (CAD/CAM) technology has enormous advantage on manufacturing precisely restorations. Reducing the distortions caused by standard methods, also eliminating numerous laboratorial phases which could be complicating factors during the manufacture process [9,11]. Dental ceramics and composites could be easily fabricated with CAD/CAM technology, with desirable properties due to adequate environment where it is produced [12,13].

CAD/CAM technology provides powerful restoration and conservative preparation, however, for best performance the materials choosen should be elected during the treatment planning [8,12,13]. Studies have shown unexpected behavior like higher fatigue resistance of composites materials when compared to ceramics. Probably due to flexural modulus be very similar to dentin, which creates a single body in terms of mechanical behavior, as consequence a wide spread of materials possibility [12,14,15].

A clear gap between CAD/CAM materials machinability interactions – which is calculated by quantitative measures with the concept of brittleness index – and their characteristics like marginal fit after mechanical tests, such as, static and dinamic loading is missing and indispensable to clinical performance conclusions [13,16]. However, the influence of fatigue and discrepancies on marginal integrity are reported for all-ceramic and gold restorations, with acceptable margins [5,12,15,16].

The first mention in literature for brittleness index [17,18], combined the results with marginal chipping factor (CF) which can be calculated only after the crowns were milled. As clearly results, ceramics have higher brittleness index when compared with composite materials, however, some materials are no longer available and new materials shown up in the recent years. Furthermore, tests which predict the brittle of a material before milling could be more relevant for clinicians.

The data sheet of LAVA Ultimate, bring the composition as 80% by weigth of ultrafine particles of zirconia and silica, which reinforces the polymer matrix. IPS e.max CAD composition is almost 60% of lithium disilicate glass ceramic (SiO_2), among others oxides. Analyzing the relation between these two materials, IPS e.max CAD have a flexural strength of 360 MPa and elasticity modulus of 95 GPa, while LAVA Ultimate has flexural strength among 150 MPa and flexural modulus of 12 GPa. The chemical composition of CAD/CAM materials tested in previous studies [18] raised the hypothesis that CAD/CAM blocks of composite resin may have a better clinical performance than ceramics from laboratory experiment. However, it is necessary a deep analysis among others aspects of mechanical properties. Exactly in this gap, our investigation is inserted, considering the

Brittleness index as newly interest aspect of aesthetics materials, and your possible relationship with others properties.

Different ceramic materials compositions affect their mechanical properties, such as; flexural strength, fracture toughness, fracture resistance, hardness, as well as brittleness index which is our main property to be investigated. The CAD/CAM technology bring in new concepts and necessity of new methods to predict the longevity of restorations in terms of susceptibility to cracks, voids and flaws; once they are machinable materials. The aim of this study is to measure the brittleness index and Vickers hardness of several CAD/CAM materials used with the CEREC system and assesses their relationship to better understand the behavior. The null hypothesis is that there is no correlation between these two properties and as the Vickers hardness increases so does the brittleness index.

2. Materials and methods

2.1. Sample preparation

Fifty-two specimens from CAD/CAM ceramic materials were selected after crowns were milled in CEREC MC XL milling unit (Sirona, Bensheim, Germany). The blocks used after milling the crowns were divided into four groups (n=13 each). The materials selected for the research were: Vitablocs Mark II (Vita Zahnfabrik, Essen, Germany) as a control group, IPS e.max CAD – intermediate phase (Ivoclar Vivadent, Schaan, Liechtenstein), IPS Empress CAD (Ivoclar Vivadent, Schaan, Liechtenstein), and the resin nano ceramic LAVA Ultimate (3M ESPE, St, Paul, USA). The materials compositions and the flexural strength provided by the manufacturer are in **Table 1**. The blocks surface was regularized using a slow-speed diamond saw (Isomet 4000, Buehler, Lake Bluff, IL, USA), and all blocks had an approximate width of 0.6mm of ceramic thickness **(Figure 1).** With the specimens surface planned, using a grinding and polishing device (Metaserv 2000, Buehler, Lake Bluff, IL, USA) equipped with sandpaper water (3M ESPE, St. Paul, MN, USA), the specimens were sequentially polish with #180, #250, #360, #400, #600, #800, #1200, and #1500. In order to create the ideal necessary smooth surface for proper data acquisition by the microhardness machine, specimens were additionally treated with a sequence of a 1μm and 0.5μm polish diamond paste (Buehler, Lake Bluff, IL, USA) in combination with the #1500 grit water sandpaper. Specimens were then cleaned by ultrasonic water bath (Boekel Analog Model 139400, PA, USA) for 15 minutes and air-dried.

Figure 1 – CAD/CAM blocks with approximate width of 0.6 mm.

Table 1 – Compositions and flexural strength of CAD/CAM materials used.

Materials	Compositions	Flexural Strength	Lot Number
Vitablocs Mark II	Feldspathic ceramic	154 MPa	33410
IPS e.max CAD	Lithium disilicate glass-ceramic	130-150 MPa	T15004
IPS Empress CAD	Leucite-based glass ceramic	160 MPa	T32757
LAVA Ultimate	Nanoceramic silica and zirconia fillers	150 MPa	N678317

2.2. Measurement of Vickers hardness and Brittleness index

For both tests, each specimen surface was divided in four quadrants. On each quadrant, several 49N load (P) indents with loading time of fifteen seconds were made using a Vickers hardness machine (W-402MVD Buehler – Lake Bluff, Illinois, USA) at room temperature, totaling 8 measures per sample and 104 per material. Only two acceptable indents per quadrant were selected for final measurement using the following criteria: *brittleness* (1) all cracks emanated from the corners of indent; (2) presence of only four radial cracks; (3) no crack chipping and; (4) no crack branching; *Vickers* (a) clean and visible indents; (b) no chipping around diamond indents **(Figure 2)**.

Brittleness index was calculated by measuring the crack along the vertex of the indentation on the same direction of the diagonal 1. Once the cracking growth is time dependent, readings were made after 30 seconds. Using the microhardness tester, two-lines markers visible on the lens were positioned on both ends of the crack. Measurements are shown in the **Figure 2** and calculated by equation 1 [19]:

Equation (1) – $B = \gamma P^{-2/4}(C/\alpha)^{3/2}$

On the equation (1) above, B is the brittleness index in $\mu m^{-1/2}$; P is the indentation loading (N), γ is the constant value equal to $2.39 N^{1/4}/\mu m^{1/2}$; C is the diagonal one and; α is the crack length diagonal one in µm.

Vickers hardness was calculated by measuring the diagonal 1 and 2 distances and analyzed by the micro hardness software on the tester machine.

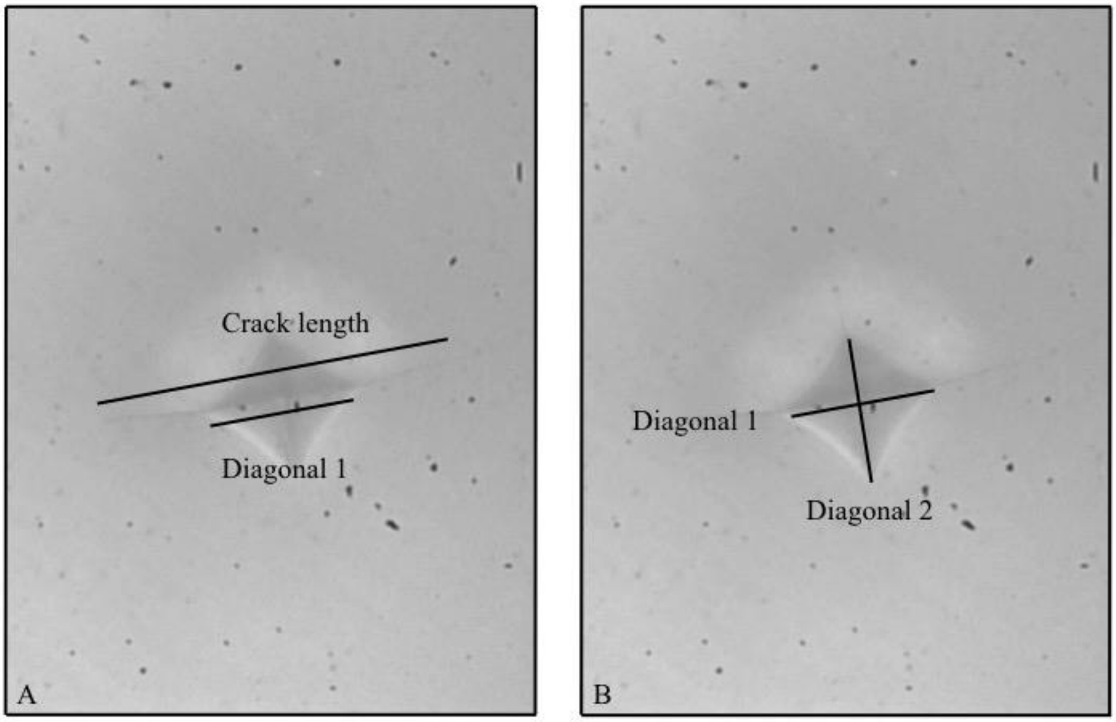

Figure 2 – Left side A – scheme of Brittleness index measurement. Right side B – scheme of Vickers hardness diagonals measurement.

2.3. Data analysis

Statistical data were calculated by SPSS 22.0 software. Levene's test and Shapiro-Wilk test was performed to make the assumptions of homogeneity variances and data normally distributed. One-way ANOVA analysis was performed followed by Games-Howell *post hoc* comparisons to find any statistically significant differences among Vickers hardness and Brittleness index accordingly with the four materials. Finally, the Pearson's correlation was calculated to analyze the interaction between both mechanical properties. The significance level was set at α=0.05.

3. Results

The statistic analysis associated with materials Vickers hardness and comparing the four different types of materials are reported in **Table 2**. A Levene's test for homogeneity **(Table A2)** of variances was used ($p<0.05$) and not satisfied, also Shapiro-Wilk's test **(Table A1)** ($p<0.05$) and a visual inspection of their histograms, normal Q-Q plots and box plots showed that the micro hardness of each ceramic are normally distributed, with a skewness of -0.316 (±0.616) and a kurtosis of -0.765 (±1.191) for IPS e.max CAD; a skewness of -0.885 (±0.616) and a kurtosis of 1.098 (±1.191) for Empress CAD; a skewness of 0.193 (±0.616) and a kurtosis of -0.665 (±1.191) for Vita Mark II; and a skewness of 1.384(±0.616) and a kurtosis of 1.694 (±1.191).

Table 2 – The descriptive statistics for Vickers hardness across the materials type.

	N	Mean*	Std. deviation	Variance	Skewness	Kurtosis
IPS e. max CAD	13	697.71^{a} Hv	16.50	272.22	-0.316	-0.765
Vitablocs Mark II	13	609.28^{b} Hv	22.44	172.63	0.193	-0.665
IPS Empress CAD	13	585.83^{b} Hv	20.94	438.58	-0.885	1.098
LAVA Ultimate	13	102.81^{c} Hv	6.50	42.37	1.384	1.694

* Same letters means that not statistically significant difference was found.

For the Vickers hardness test, one-way ANOVA **(Table A3)** was performed and showed statistically significant differences among the samples ($p<0.05$). Games-Howell *post-hoc* test **(Table A4)** showed statistically significant difference among ceramic materials by multiple comparisons ($p<0.05$). The IPS e.max CAD group (697.71±16.50) was significantly higher than all other groups, while Vitablocs Mark II (609.28±22.44), was not significantly different from IPS Empress CAD (585.83±20.94), and LAVA Ultimate (102.81±6.50) was significantly lower than all other groups.

The brittleness index testing for each ceramic group was calculated based on the measurement of the loading indentation and cracking along the vertex. The descriptive statistics with materials Brittleness index are reported in **Table 3**. A Levene's test for homogeneity of variances was applied ($p<0.05$) and not satisfied; also Shapiro-Wilk's test ($p<0.05$) and a visual inspection of their histograms, normal Q-Q plots and box plots showed that the micro hardness of each ceramic normally distributed, with a skewness of -0.250 (±0.616) and a kurtosis of -0.796 (±1.191) for IPS e.max CAD; a skewness of 0.459 (±0.616) and a kurtosis of 1.545 (±1.191) for Empress CAD; a skewness of 0.053 (±0.616) and a kurtosis of -1.065 (±1.191) for Vita blocks Mark II; and a skewness of 1.265 (±0.616) and a kurtosis of 3.067 (±1.191) for LAVA Ultimate.

Table 3 – The descriptive statistics for Brittleness Index ($\mu m^{-1/2}$) across the materials type.

	N	Mean	Std. deviation	Variance	Skewness	Kurtosis
IPS e. max CAD	13	2.53[a]	0.17	0.029	-0.250	-0.796
Vita Mark II	13	2.31[a]	0.11	0.011	0.053	-1.065
IPS Empress CAD	13	1.77[b]	0.11	0.013	0.459	1.545
LAVA Ultimate	13	0.91[c]	0.03	0.001	1.265	3.067

[*] Same letters means that not statistically significant difference was found.

One-way ANOVA was also performed for Brittleness index and showed statistically significant differences among the materials ($p<0.05$). Games-Howell *post-hoc* test showed statistically significant difference by way of multiple comparisons ($p<0.05$). The IPS e.max CAD (2.54±0.17) was not found to be statistically significant different than Vita Mark II (2.31±0.11), however both materials were statistically significant higher than IPS Empress CAD (1.77±0.11) and LAVA Ultimate (0.91±0.03).

Once Vickers hardness and Brittleness index are both dependent variables, Pearson's correlation was used and showed statistically significant relationship between both dependent variables ($p<0.05$). There is a significant positive relationship involving Vickers hardness and brittleness index ($r\,[50] = 0.93$) **Figure 3.**

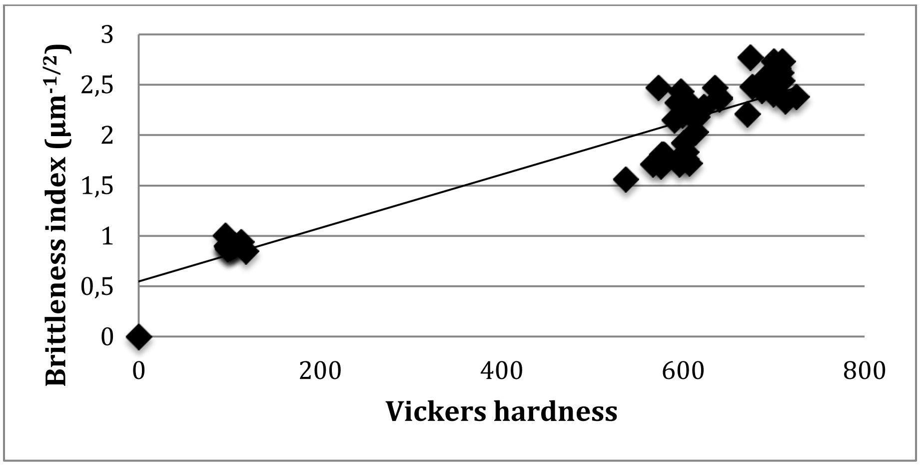

Figure 3 – Pearson correlation between Vickers hardness (x) and Brittleness index (y).

4. Discussion

The purpose of this study was to investigate and evaluate the Brittleness index and Vickers hardness of different CAD/CAM materials and also the correlation between both measurements. The developments of CAD/CAM technology combined with the newly ceramic materials specific designed for this technology are awakening the curiosity of many researchers [20-22]. The principles of chair-side CAD/CAM are becoming increasingly overspread among clinicians and universities, changing the common way of learning indirect restorations, and providing the access to more people apply them in practice [23-25]. A new skill is starting to be as major importance for future dentists, competence and ability to do digital restoration. Furthermore, the mechanical properties of recent and newly CAD/CAM materials demand from clinicians, knowledge and familiarity [18]. The behavior of a variety of CAD/CAM ceramics is introduced in new concepts; as materials machinability, which is still not so well defined. The combination of inappropriate preparation design as well as the chosen materials can decrease the restoration longevity.

The first null hypothesis was that there is no correlation between Vickers hardness and Brittleness index, although, the hypothesis was rejected. As a result from the Pearson's Correlation Coefficient a relationship of 0.93 was achieved, meaning a strong relationship between Brittleness index and Vickers hardness, with a magnitude of 86%. Thus, the use of hard material means more susceptible to chipping, because the machinability of the materials with high Brittleness index is poor than the ones with lowers values. Ceramics made of lithium disilicate showed the highest values of Brittleness index as well as the Vickers hardness, while the IPS Empress CAD a feldspar leucite-reinforced glass ceramic show intermediate values. As expected the LAVA Ultimate a composite resin CAD/CAM block presented the lowest values, and can be considered a material with the best machinability among the ones tested. Based on the results of this study, the composite resin CAD/CAM materials will perform better in matters of machinability when compared with ceramic materials; and is reliable to consider that materials with high values of Vickers hardness will perform badly when they are supposed to be used combined with CAD/CAM technology.

The first research to discuss the relationship of Brittleness index and others CAD/CAM mechanical properties [18] have raised some interesting questions among the use and the consequences of machining process. The proposal of Chipping Factor is reliable and can predict the materials machinability, however, can only be measured after milling [18]. Explore the relationship against other mechanical properties will lead the researchers to better understand the Brittleness index as well as the behavior of CAD/CAM ceramics. Despite the fact that the appearance of surface imperfection due to milling process, materials with high values of Brittleness index should not be used in restorations that require principles of minimal invasive dentistry. Our research showed that the materials hardness can be another predictable of machinability with a great advantage, a simply inquiry on companies materials leaflet can teach the clinicians and lead them to chose the appropriate material. CAD/CAM technology needs easy and simple prediction of machinability, factors to predict like tool wear, chipping control, machining and energy forces, and surface finish and integrity unfortunately can only be checked after manufacturing process and are qualitatively analyses [20]. Due to application of the modern way of manufacturing ceramic restorations, quantitative analyses is extremely necessary in terms of dealing with the new materials purpose.

A recent research of fracture resistance calculated by the thickness of the CAD/CAM [12] mimic designed restorations for posterior tooth has shown results that are comparable with our findings. The fracture resistance of LAVA Ultimate was higher than the IPS e.max CAD at a thickness of 0.5mm; however, when the thickness was increased to 3.0mm they had the opposite as result [26], with strong positive linear relation between the thickness and fracture load for IPS e.max CAD. Combining both findings, a material with the highest level of Brittleness index, as well as low performance on fracture resistance at thin thickness must have specific indications. Despite that fact it is still one of the best materials when used correctly. Our results shown that the materials with highest Vickers hardness are the most sensitive to fracture, and previous studies mentioned the fracture resistance being affected by occlusal thickness of ceramic and composite crowns [27-28], this might lead us to define when soft or hard materials should be used in clinical situations. Comparing the occlusal reduction ranges recommended that goes from 1.3mm to 2.0mm [15, 27-29] with the results of Brittleness index, materials with higher index must be indicated when enough tooth reduction are well satisfied, without the risky of premature failure.

The milling process of CAD/CAM technology can cause inner flaws, voids and cracks; as we can see on **Figure 4** the concentration of loading around a pre-crack is much higher in ceramics than composite, furthermore the literature imply as a possibility radial cracking mode for thin ceramics [29-31]. Bulk properties shall be significant for ceramic materials only at 2.0mm of thickness [26], however, because of such great mechanical properties dental ceramics are allowed to be use below that thickness.

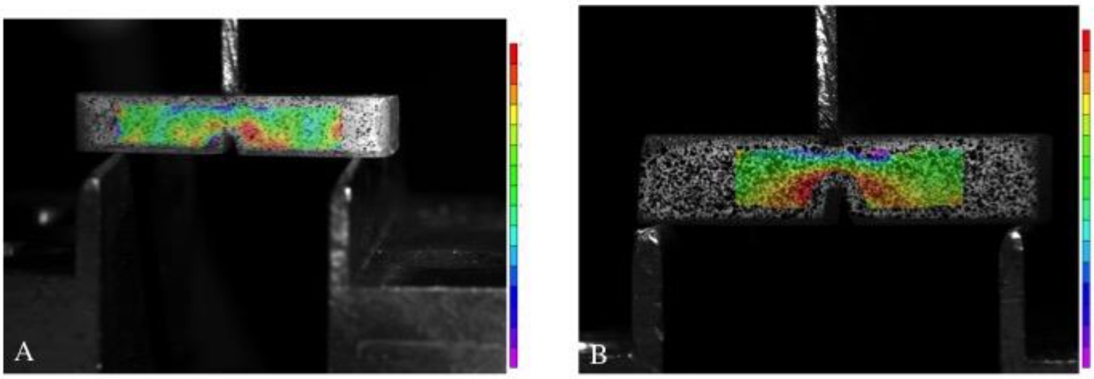

Figure 4 – A: Loading a ceramic beam sample to complete failure to check the loading distribution. B: A CAD/CAM composite resin block under loading to check the loading concentration.

Hardness is defined as the resistance to a permanent surface indentation, as consequence ceramics hardness impact on polishing procedures, wear resistance and machinability [32], also the brittleness index has a strong positive relationship with that property causing to be one of the most important features to be analyzed before choosing a restorative CAD/CAM material.

5. Conclusion

Currently, CAD/CAM restorations are not always successful, because of overall materials characteristics and poor indication; it is a continual difficulty (issue) that every clinician is faced with. In this study, we found that an important mechanical property, such as the brittleness index can make a real difference into the day-by-day clinical application of these materials. Our findings should be eventually tested with different *in vitro* experiment, such as crown fracture resistance, in order to develop a better understand of the materials behavior.

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3.2 Publicação 2

Title: *Effect of thermal cycling on fracture toughness of CAD/CAM materials**

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Keywords: ceramics, dental porcelain, CAD/CAM, chipping, fracture load, fracture toughness, glass ceramic

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Abstract

Objectives: The purpose of this in vitro study was to compare the fracture toughness under long thermo cycling conditions of four different CAD/CAM materials, Vitablocs Mark II (G1), IPS e.max CAD (G2), LAVA Ultimate (G3), and Vita Enamic (G4).

Methods: Each type of ceramic block was sectioned into beams (n=9) with dimensions of 14x3x2.5mm (L x W x H). All 4 ceramics were submitted to different conditions: the first condition was into the desiccator; the second in distilled water, both for 7 days. On the third and fourth condition the specimens were submitted to thermocycling in 60.000, and 120.000, respectively. The dwelling time was 52 seconds (5^{o} and 55^{o}C). A three-point bend test with a universal loading machine on notched samples was performed. Furthermore, on fractographic analyses were made on SEM to determine if the conditions had influence on the type of fracture. Data were analyzed by two-way ANOVA (α=0,05).

Results: A significant difference in fracture toughness ($P<0,05$) was found among groups, G2 (0,705±0,10) had the highest value followed by G4 (0,478±0,10); which did not have significant statistically difference from G3 (0,467±0,10), and with the lower value and statistically different, we had G1 (0,0399±0,10). The storage conditions shown the ceramics performing better after they were hydrated, and the materials with composite in their formula had shown worst performance.

Conclusions: The fracture toughness of VM shown the best performance after 120.000 cycles, showing as further as the material remains in function better shall be. The materials with composite in their formula will be degraded with time.

Clinical Significance

The clinicians should pay attention to the particularities that they would be faced with, based on the materials chosen for every case. The research showed important features related to each class of CAD/CAM materials.

1. Introduction

Over the last two decades, important improvements involving ceramic mechanical properties have occurred. However, the ceramics are still considered a restorative material with low tensile and flexural strength, making the material brittle and sensitive to cracks and flaws. In this context, some researchers have demonstrated a higher incidence of failure of these materials [1,2]. The use of computer-aided design and computer aided manufacturing (CAD/CAM) create the possibility to mill ceramic restorations from a solid block as an alternative of making with conventional multiple firings or even by the pressable system. Ceramic restorations made from a solid block might present reduced flaws and defects, as well as improved mechanical properties compared to conventional manufacturing ways [3].

The very first CAD/CAM restorations were generated from fine grain feldspathic ceramic (Vita™ Mark I) with a flexural strength around 120 MPa and they were only indicated for fabrication of inlays, onlays and veneers [4-6]. The Vitablocs™ Mark II was introduced in 1991 with better mechanical properties in terms of flexural strength (160 MPa) [7]. Noted improvements in mechanical properties for Vitablocs™ Mark II blocks were due to the extrusion molding of the "traditional" feldspathic material, whereas a plasticized ceramic mixture was pushed through a die with subsequent drying and sintering processing [4,6,7].

Lithium disilicate is another glass ceramic that has been used in dentistry since 1998. It was first introduced through the “lost-wax” technique for single tooth restoration and later overhauled in 2001 named IPS Empress 2 (Ivoclar Vivadent, Schaan, Liechtenstein) with enhanced features and higher survival rates [3,8]. The IPS Empress 2 essential characteristics are based on lithium disilicate crystals measuring approximately 3 to 6μm in length, and 70% needle-like crystals, which are embedded in a glassy matrix [9]. A new version of IPS Empress 2 was introduced during the year of 2005 as IPS e.max Press with a flexural strength of 400 MPa, for laboratory technique and IPS e.max CAD with 360 MPa for CAD/CAM purposes [10]. The unusual purple color of CAD/CAM blocks for lithium disilicate is due to a crystalline intermediate phase, which is provided by 40% platelet-shaped lithium metasilicate crystals and glassy matrix [10].

The CAD/CAM restorative materials are mainly composed of ceramics and resin-based materials [1,11]. Composite CAD/CAM restorations might not be as hard as ceramic, suffering with material wear. Contrarily, ceramic CAD/CAM restorations are reported to be very abrasive against antagonist dentition [1]. The ideal restorative material has not yet been established; therefore developing of new CAD/CAM materials are still ongoing. Recently, a new class of CAD/CAM blocks has been introduced into the market, so-called hybrid resin nano-ceramic. The Vita Enamic block (Vita Zahnfabrik) is a polymer infiltrated ceramic network with a flexural strength of 155 MPa, according to manufacturer's information. The LAVA Ultimate (3M ESPE, St Paul, MN USA) a nanoparticle and nanoclulster-filled resin also presenting a flexural strength around 155MPa. Polymer-based materials are well-known for having more elastic deformation before fracture compared to ceramics. An organic polymer matrix supported by inorganic filler particles, such as silica-based, allow the clinical performance of hybrid materials to be less brittle and more flexible [1]. However, recent studies have shown that different flexural strength for the same material [1,12-14], once that the fracture resistance of all-ceramic restorations is influenced by different factors such as, the mechanical properties, sample preparation and surface characteristics [15]. The choice on preparing the intaglio surface for cementation may also affect the surface microstructure and lead to substantial reductions on the mechanical properties [16,17].

Despite the fact that the ceramic mechanical properties have been improved significantly, the material remains sensitive to cracks and flaws. This brittle behavior associated with the intraoral conditions (mechanical and thermal) might lead the ceramic to premature failures. Various studies have been focused around flexural strength, defined as a materials ability to resist deformation under load [1,4,12,13]. Relatively few studies focus on fracture toughness (Kic), which can better describe the ability of a material to resist to fractures by containing a crack and consequently, a catastrophic failure. The Kic factor is also one of the most important properties of any material for many design applications [18] and appears to be more relevant to fracture of ceramic and hybrid materials than flexural strength. Hybrid materials, contrarily, are fabricated by a process involving a ceramic network conditioned by a coupling agent infiltrated with a polymer. The ceramic network is composed by two main components; a feldspar phase and a zirconia crystalline phase, both responsible to increase the strength of hybrid materials [11]. The polymer used to infiltrate the ceramic network is made of organic matrix phase. It is unclear if hybrid materials are susceptible to water absorption, which would cause hydrolysis on the interfacial polymer network [13]. Recent research demonstrates that microcracks findings around the ceramic network might decrease the fracture toughness of dental materials [11] thus with a potential hydrolysis process that may lead this materials to premature failures. Consequently, the aims of the present study are; i) to perform thermal cycling processes in both ceramic and hybrid CAD/CAM materials in order to determine the fracture toughness ii) fractographic analysis of different fracture types on both materials. The null hypotheses are that (i) thermal cycling conditions would have no significant effect on K_{ic} values for all types of materials and; (ii) different materials will have significant effect on K_{ic} values.

2. Materials and Methods

Four different CAD/CAM blocks (all size I14) were designated for the study purpose: Vitablocs Mark II (Vita Zahnfabrik, Essen, Germany) as a control group (G1), IPS e.max CAD (Ivoclar Vivadent, Schaan, Liechtenstein) as group two (G2), LAVA Ultimate (3M ESPE, St Paul, MN, USA) as group three (G3) and Vita Enamic (Vita Zahnfabrik, Essen, Germany) as group four (G4). Details about each block composition and batch numbers are listed in **Table 1**. Specimens were fabricated from each CAD/CAM block by firstly sectioning the blocks into slices (14.8mm x 12mm x 3mm) and then into beam shaped specimens (14.8mm x 3.5mm x 3mm), by means of a precision cutting machine (Isomet 1000; Buehler, Lake Bluff, USA). To avoid ceramic chipping during the cutting procedure and to achieve a more precise dimension, specimens were submitted to finishing and polishing procedures (Ecomet 6, Buehler, Lake Bluff, IL, USA) using the following sequence of sandpapers: 240, 400, 600, 800 and 1200 grit. Final dimensions (14mm x 3mm x 2.5mm) were confirmed by a digital caliper (Mitutoyo America Corporation, Illinois, USA).

Table 1 – Material commercial name, manufacturer, composition and batch number.

Material	**Manufacturer**	**Composition**	**Batch**
Vitablocs Mark II	Vita Zahnfabrik	SiO_2, AL_2O_3, Na_2O, K_2O, CaO, TiO_2, coloring oxides	51410
IPS e.max CAD	Ivoclar Vivadent	SiO_2, Li_2O, K_2O, P_2O_5, ZrO_2, ZnO, Al_2O_3, MgO, coloring oxides	27280
Vita Enamic	Vita Zahnfabrik	SiO_2, AL_2O_3, Na_2O, K_2O, B_2O_3, CaO, TiO_2, coloring oxides, methacrylate polymer	47791
Lava Ultimate	3M ESPE	SiO_2 (20nm), ZrO_2 (4-11nm), nanoclusters	N676551

2.1 Fracture toughness (K_{ic})

The specimens were positioned with 2.5mm wide face up, to be notched in a plastic ring with 16mm diameter and 10mm height filled with premium dental stone (Golden Type 3, Whip Mix, Louisville KY, USA) to hold the beam samples. All one hundred and forty four beams were set into the plastic ring. Positioned in a holding device the ensemble were attached by clips to X-axis, on the Y-axis was connected a hand-piece (Henry Schein, Melville, NY, USA) with a needle shape diamond point 862-016FC (SS White, New Jersey, USA). The V-notch was created using the diamond point along the Z-axis; circular screw attached to all three entire axes has a control of 0.01mm. The notch depth was approximately to 1mm. The beam specimens positioned into the plastic ring were carefully removed and cleaned using deionized water in a sonic bath for 30 min. The notch size of each specimen was accurately calculated; the fracture part was measured after the three-point bending test with a digital caliper and subtracted from the width (3mm).

Following the ASTM C1421-10, the V-notch relation was obtained through the equation (1) $\alpha = a/W$ and the ratio were approximately to 0.32. Specimens were positioned with flat surface facing up and the V-notch centered on the supporting spam. The three-point bending test for K_{ic} calculation ran at a crosshead speed of 0.5mm/min and with a support span (S_0) of 12mm in a universal testing machine (Test Resources, Shakopee, MN, USA) until failure at a room temperature (23±2^{o}C). The equation (Eqs (2) and (3)) used for K_{ic} calculation in V-notched beams was set to be used in Microsoft Excel for Mac 2011 (version 14.5.8).

$K_{ic} = g[PmaxS_0 10^{-6} / BW^{3/2}] [3(a / W)^{1/2} / 2(1-a / W)^{3/2}$ (2)

$g = [1.99 – (a / W)(1 – a / W)\{2.15 – 3.93 (a / W) + 2.7 (a / W)^2\}] / [1 + 2 (a / W)]$

(3)

In the equation (2) K_{ic} is fracture toughness (Mpa.m$^{1/2}$), *g* is a function of the ratio a/W, *Pmax* is maximum fracture load (N), S_0 is the distance between the center of the rollers (m), B is the thickness of the test specimen parallel to the support rollers (m), W is the width perpendicular to the support rollers (m), and *a* is depth of V-notch (m). The equation (3) was used to calculate the *g* function based on *a/W* ratio.

2.2 Thermo cycling

The initial four testing materials were further divided into four subgroups (n=9) according to the conditions that specimens were storage. The thermo cycling process was made with Thermo Haake (Thermo Haake, Karlsruhe, Germany), each cycle lasted for 52 seconds, as follows, the dwell time within each bath (55^o and 5^{o}C) was 20 seconds, and the transferring time between baths was 6 seconds.

- Condition A: 7 days in desiccator at room temperature; no thermo cycling
- Condition B: 7 days at 37^{o}C in distilled water; no thermo cycling
- Condition C: 60,000 thermo cyclers (between 5^o and 55^{o}C) within 40 days
- Condition D: 120,000 thermo cyclers (between 5^o and 55^{o}C) within 80 days

2.3 SEM analysis

All beam-shaped specimens were first analyzed through an optical microscopy (35x magnification, Nikon SMZ445, Melville, NY, USA) to identify the type of fracture, crack path and features. The most relevant specimens were designated to SEM (FEI Nova Nanolab 200, Hillsboro, OR, USA) analysis and fractographic evaluation. (ASTM c1322-05b standard practice for fractography and characterization of fracture origins in advanced ceramics).

2.4 Statistical analysis

The statistical package SPSS 23.0 was used to analyze the data acquire from the study. The data was first submitted to homogeneity and normality tests, although, they were not satisfied. Hence, the data were log transformed to achieve minimal requirements of parametric analysis. Two-way ANOVA was performed to find statistically significant differences among the materials, and conditions. Finally, Tukey's *post-hoc* tests were performed to identify where the differences occur.

3. Results

The data obtained during the fracture toughness test was submitted to log transformation, in order to obtain the requirements for two-way ANOVA analysis **(Table A5)**. The original histogram a slightly positive skewness was observed, however, after the log transformation a normal distribution was achieved. The means comparison summarized in **Table 2** from ceramic and hybrid materials were means obtained through three-point bending test and already log transformed, also standard error, variance and skewness are provided.

Table 2 – Means (standard error) of fracture toughness (Mpa.m$^{1/2}$) related to CAD/CAM materials.

	Mean log10 (Std. Error)	Variance log10	Skewness log10 (Std. Error)
IPS e.max CAD	0,705 (0,10)[a]	0,009	0,548 (0,393)
Vitablocs Mark II	0,399 (0,10)[b]	0,004	0,259 (0,393)
LAVA Ultimate	0,467 (0,10)[c]	0,004	-0,590 (0,393)
Vita Enamic	0,478 (0,10)[c]	0,003	0,069 (0,393)

[*]Means with same letters were not considered different at the significance level.

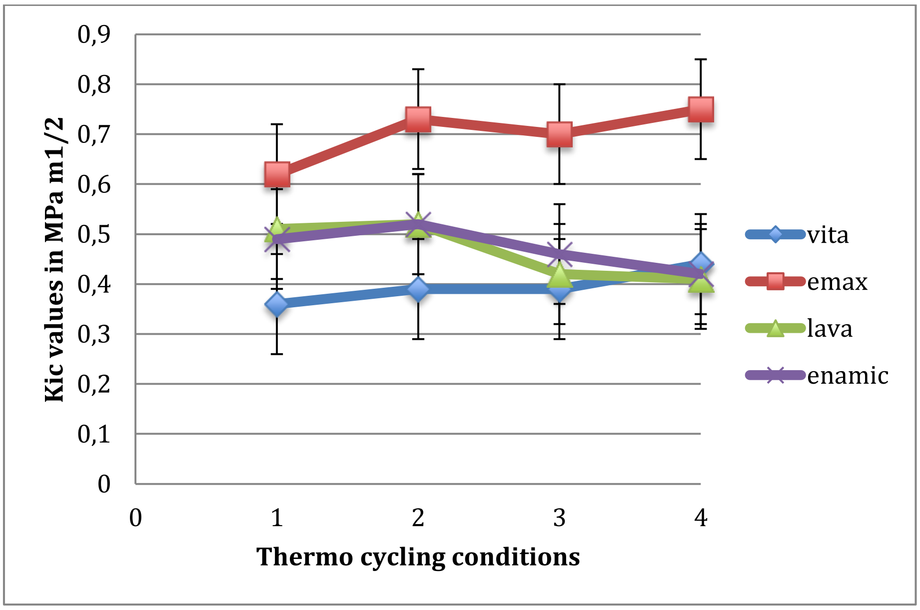
The conditions that the CAD/CAM materials were submitted shown an increase in fracture toughness for G3 and G4 **(Figure 1)** after they were water storage for one week, when compared against dry condition. However, after thermo cycling process the poorest fracture toughness resistance was observed, but no statistical significant difference was determined between 60.000 and 120.000 **(Figure 1)**. Our results reinforce the hypothesis that after sometime the water absorption would cause hydrolysis of the interfacial silane-coupling agent, and the hybrid materials will behavior similar to composite (see discussion).

The ceramic materials had a completely different behavior, however, very similar between each other. Under the dry condition the poorest fracture toughness resistance was observed, nevertheless after one week of wet condition, without statistical significant difference an increase of the means for G1 and G2 were observed **(Figure 1)**. G1 shown only after 120.000 cycles, an increase of the mean with statistical difference among other conditions, suggesting that the material would perform better in terms of fracture resistance along the time. G2 had only statistical significant difference **(Table A6)** between the dry condition against the other three wet conditions. A hydration of the material may be an important preoperative procedure before the cementing process.

Table 3 – Means (standard error = 0.28) of fracture toughness (Mpa.m$^{1/2}$) related to CAD/CAM materials and thermo cycling conditions.

Group	Desiccator	Water storage	60.000 cycles	120.000 cycles
Vitablocs (G1)	0.36^{a1}	0.39^{a1}	0.39^{ab1}	0.44^{b1}
IPS e.max (G2)	0.62^{a2}	0.73^{b2}	0.70^{b2}	0.75^{b2}
LAVA (G3)	0.51^{a3}	0.52^{a3}	0.42^{b13}	0.41^{b1}
Enamic (G4)	0.49^{ab3}	0.52^{a3}	0.46^{bc3}	0.42^{c1}

*For each horizontal row: values with same letters indicate no statistically significant difference (p>.05)
**For each vertical column: values with same numbers indicate no statistically significant difference (p>.05)



*1 = dry condition / 2 = water storage / 3 = 60.000 cycles / 4 = 120.000 cycles

Figure 1 – Material performance in comparison against each other and conditions applied.

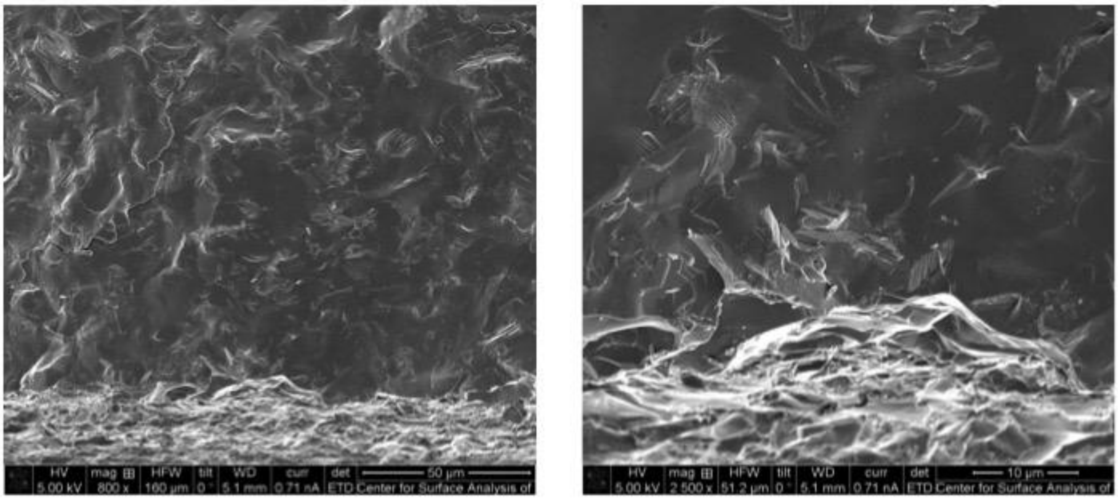


Figure 2 – Vitablocs Mark II – At the bottom of the picture is possible to identify the fracture origin, with pore at the 800 x magnification picture, also a mirror region with mist and hackle region characterizes the surface.

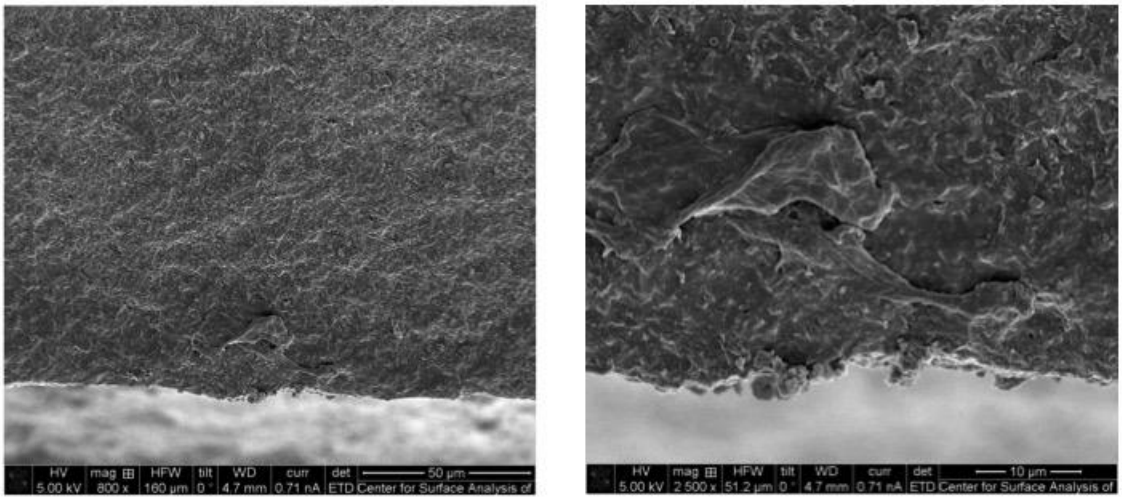


Figure 3 – IPS e.max CAD – Due to lithium disilicate crystals like needle orientation it is not visible mirror region. Flaws mist and hackle region characterize the fracture origin at the bottom of the pictures. Porous region which identify 3-dimensional zone of porosity and microporosity is visible at the 800x magnification picture.

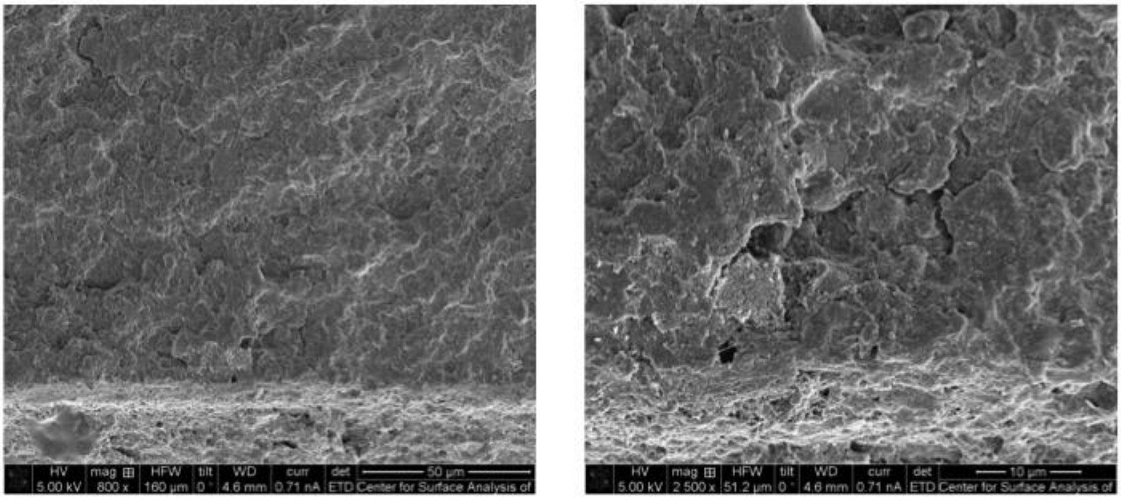


Figure 4 – Lava Ultimate – The surface of this resin nano ceramic material shows coarse hackle, flaws along the surface and in both pictures the entire surface is dominated by a hackle regions. Also large grains are apparent in the middle of the 800x magnification picture.

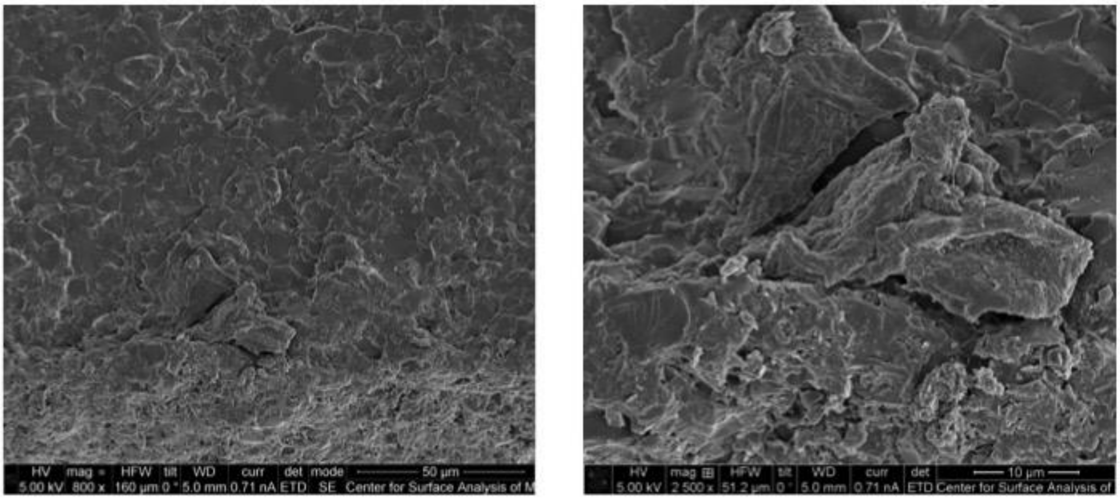


Figure 5 – Vita Enamic – The ceramic hybrid material besides the fact that has a similar behavior to composite materials after the mechanical tests, the fractrographic analysis shows regions of brittle fracture, flaws, grains boundary, and also porous region; which characterize the material with a ceramic behavior.

4. Discussion

The null hypotheses which states that (i) thermal cycling conditions would have no significant effect on K_{ic} values for all types of ceramics was rejected; and (ii) different ceramics will have significant effect on K_{ic} values was accepted. Within the limitations of this simulated long thermal cycling study, the materials behavior were different, with an exception for Lava Ultimate and Vita Enamic; not coincidentally the materials with polymer in the composition that are shown in **Table 1** had not statistically significant difference in K_{ic} values. Besides resin-based composites are formed by organic polymer matrix and inorganic fillers such as silica, zirconia and barium [1], Vita Enamic possess a ceramic-network, which is infiltrated by polymer [11], while Lava Ultimate present inside the material composition, nanocluster particles such as; silica and zirconia, which provides structural integrity allowing a high quantity of ceramic filler. Known as polymer-infiltrated-ceramic-network and resin nano ceramic respectively, both had similar behavior without considering the conditions where they were submitted.

The fracture toughness (K_{ic}) known as the property of a material to resist crack propagation has been reported as one of the key features connected to clinical performance [11,19,20]. Ceramics known as brittle materials had different reaction under the three-point bending test developed during this study. IPS e.max CAD demonstrated the best performance, while Vitablocs Mark II had the worst, compared with materials along this research protocol. The condition of thermo cycling process states the possibility of *in vitro* simulation of the mouth environment related to terms as moisture and temperature alteration. So far studies had only simulated fatigue and resistance alone or associated with thermo cycling process [1, 11, 12, 17, 18, 21-26]; however, the materials behavior had always been related to mechanical procedures, and just a few research implied the action of moistures conditions in ceramics or polymers CAD/CAM materials [13].

The influence of moistures environment on ceramic mechanical properties definitely are unknown, previous studies always related the failures with mechanical and thermo cycling loading combined; and when they are not joined into one experiment only mechanical loading is reported. Thermo cycling is most used to report the effect on bond strength of luting cements and adhesive procedures [27, 28], whereas is also known the influence of water absorption on composites [29] and recently reported in composite CAD/CAM materials [13]. Previous research had demonstrated [30,31] residual stress on composite, and even ceramic that may influence the mechanical properties. On our samples the three point bending test worked as a compressive strength on the side, which the load was applied, whereas underneath the sample where the notched was located it was a tensile strength. On the manuscript of Taskonak [32] the mention to increasing strength while compressing and decreasing while tensile was not completely found in this research.

IPS e.max CAD showed a different behavior taking upon recent researches, considering that they focus on mechanical properties after fatigue and different types of surface treatments. The lithium disilicate had demonstrated increase of fracture toughness after 7 days water storage compared to the dry condition, furthermore, after performing the thermo cycling of 60,000 and 120,000 no decrease on the fracture toughness performance was observed. Clinically speaking the findings must give an orientation of at least hydrating the lithium disilicate restoration before final cementing procedure. Vitablocs Mark II presented a very similar behavior under the same conditions; the only difference was that best performance was shown after 120,000 thermo cycles, considered statistically significant different, and temperature changes had no effect in decreasing the performance on pure ceramic materials in the long run.

No matter it was a resin nano ceramic or polymer-infiltrated-ceramic-network, both CAD/CAM materials had the same behavior after the conditions applied within this research. Once these materials are manufactured under ideal industry conditions, a superior performance would be expected, however, they still demonstrated decreasing in fracture toughness performance similar to composite resin mechanical properties [29]. Different from CAD/CAM ceramic materials after hydrating them in distilled water for 7 days, no better performance was achieved, nevertheless, a decrease on fracture toughness was demonstrated after 60,000 and 120,000 thermo cycling. Following the hypothesis raised by other authors [13], an agreement was found during this study and the water sorption would cause hydrolysis of the interfacial layer between the inorganic fillers and polymethyl methacrylate. Described with similarities between ceramic network from Vita Enamic and filler particles [11, 33-35] from resin-based composites like Lava Ultimate, microscopy high magnification evidence microcracks along the ceramic network and polymer phase [11, 35], which must be taken in consideration analyzing the water sorption, furthermore, this aspects might lead the decrease of mechanical properties.

It must be concerned that oral environment conditions are complex to replicate in terms of their contributions to the behavior of dental materials related to water sorption as well as temperature changes [13, 33]. The great amount of research published on CAD/CAM mechanical properties has improved our understanding of materials behavior and led the industry development to new hybrid materials, trying to improve their properties in terms of being very similar to natural tooth [11, 29]. However, within the limitations of our findings it seems the common weakness, regardless the materials composition and type are still present, and in means of clinical application details related to restorations procedure and use of materials should be addressed to achieve the best clinical results.

5. Conclusion

Currently, CAD/CAM mechanical properties are challenging the clinicians and the researchers, by means of understanding their behavior under real oral environment conditions. The industry development create a path based on both clinical desire as well as research experiment, however, despite of the improvements made in recent years a great amount of effort should be done to reach new levels of knowledge and experience among dentistry society. We strongly encourage new researches on the way of understanding materials behavior through their mechanical properties.

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3.3 Publicação 3

Title: *CAD/CAM materials flexural strength and flexural modulus after aging through thermo cycling conditions**

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Keywords: ceramics, dental porcelain, CAD/CAM, chipping, brittle ceramics, flexural strength, elasticity modulus, glass ceramic

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Abstract

Purpose: The aim of this study was to determine the flexural strength and flexural modulus behavior after long thermo cycling process.

Materials and methods: The materials included on this research were: Vita Enamic (Vita Zahnfabrik), Lava Ultimate (3M ESPE), Vitablocs Mark II (Vita Zahnfabrik), and IPS e.max CAD (Ivoclar-Vivadent). Thirty-six beam shaped bars (2.5 x 3.0 x 14 mm) were made for each material and divided in four subgroups (n=9): (1) desiccator for 7 days, (2) 37^{o} C for 7 days, (3) 60,000 thermocycling, and (4) 120,000 thermocycling (5^{o} and 55^{o} with dwell time of 52 seconds). The specimens were subjected to a 3-point flexural test on a 12mm span with a crosshead speed of 0.5mm/min. The results were analyzed by two-way ANOVA and *post-hoc* Tukey's test was performed for both variables.

Results: The mean of flexural strength (SD) and flexural modulus (SD) among the materials were the highest for IPS e.max CAD 396.19 (75.65) MPa and 84.97 (11.12) GPa, respectively. Lava Ultimate had the lowest flexural modulus 12.90 (3.73) GPa. From the subgroup (1) to subgroup (4) on the flexural strength the values always ranged from Vitablocs Mark II with lowest means and IPS e.max CAD with the highest ones.

Conclusion: The development of dental materials allows the recent polymer-based materials perform better than some ceramics, however, one of the major problems related to polymer-based materials still remain irresolvable. The drop of the values in flexural strength made it clear the weakness of the materials under moisture conditions.

Clinical Implications

The research revealed that ceramics in general should be hydrated before the final cementation procedures and that the thermo cycling process reduces the flexural strength of CAD/CAM polymer and hybrid materials.

Introduction

Besides the fact that computer-aided design/computer-aided manufacturing (CAD/CAM) were introduced in dentistry in middle 1980s, only recently it has gained popularity and increased worldwide. Considering an ideal restorative material with characteristics such as crack tolerance, ease of clinical use and good performance[1,2], CAD/CAM technologies can aggregate all these features. New materials have been introduced in the market recently and new ones will continue to be developed, yet there is not a perfect one[3,4]. High aesthetics requirement plus the characteristics of an ideal restorative material increase the necessity of research around very particular attributes which involves physical and mechanical properties like thermal, optical, water absorption, fracture toughness, fracture resistance, and fatigue resistance[1,2].

The CAD/CAM technology presents dentistry with an incredible advantage, eliminating the dimensional changes during the ceramics and composites laboratory manufacturing process. Despite the frequent use of ceramic, composite resin-based blocks were developed[5] based on the advantages provided by this class of material, such as higher modulus of resilience and a great flexural modulus and strength[6]. However, in order to achieve and increase these mechanical properties, fillers are an important component of resin-based materials. Industrial production allows greater quantity and volume fraction to be incorporated without affecting the mechanical durability. Furthermore, the curing degree achieved decreases the quantity of unreacted elements and increases the fatigue resistance[5].

Literature has shown tests like bond strength using luting resin-based materials are less effective after long-term thermo cycling[7,8,9] and raised the hypothesis of water absorption in CAD/CAM composite materials[10]. However, just a few studies have been found describing this problem. The filler particles are bonded to resin matrix using specific primers[11], although zirconia particles in particular suffer from debonding process under water storage[11,12,13,14], which may occur due to the difficulty of zirconia surface etching procedures. Furthermore, dental resin-based composites are dependent on the diffusion rate of free radicals and unreacted molecules inside the polymer matrix after the polymerization procedure[15]. Another important aspect resultant from the curing process is the residual stress, normally causing cuspal deflection and also marginal gap between the cavity walls[15]. As previously mentioned, CAD/CAM provides a better quality of restorations in terms of material performance and eliminates some concerns inherent to dentistry procedures. Therefore principles involving preparation to the final cementation process related to indirect procedures[16] must be followed to achieve a superior condition under sensitive procedures.

Another important aspect to be considered in dentistry procedures is the concept of microleakage, which is the passage of fluids, molecules, bacteria, or ions between the cavity wall and the restorative material. Furthermore, recurrent caries are normally associated with it, in porcelain and composite restoration[17]. These restorations manufactured through laboratory process sometimes create an inevitable variability intrinsic to the process. The CAD/CAM technology produces the ideal condition to manufacturing aesthetics restoration eliminating most part of the variability associated with the laboratory technique. Some of the clinic conditions normally associated with recurrent caries is the fracture of ceramic restoration, thus a good choice would be the repair with composite materials due to preservation of supporting structures, low cost, and fast resolution[18]. Consequently, the manufacturing companies have been using what is known about embedded composite and ceramic in the new commercially CAD/CAM materials. Repairing ceramic with composite materials has always been a challenge due to all the different mechanical characteristics. However, under ideal industrial manufacturing conditions, the CAD/CAM creates a possibility: it can fill resin-based composites with ceramic small particles such as silica and zirconia.

The purpose of this study is to address the influence of thermo cycling on flexural strength as well as flexural modulus, answering the following questions: Are the new resin nano ceramic materials and hybrid materials comparable with different ceramics as a control group? Can both classes of materials be used regardless of the treatment? Are these new CAD/CAM materials based on ideal factory manufacturing different from standard composites in terms of water absorption? Supported by these questions the objectives of this study are: evaluating differences on flexural strength and flexural modulus before and after thermo cycling and also determining whether there are influences on fractographic analysis.

Materials and methods

The current study included a broad spectrum of CAD/CAM dental materials with similar mechanical behaviors in terms of clinical indications. The materials were Vitablocs Mark II (Vita Zahnfabirk, Bad Sackingen, Germany), IPS e.max CAD (Ivoclar-Vivadent, Schaan, Liechtenstein), Lava Ultimate (3M ESPE Dental Products, St. Paul, USA), and Vita Enamic (Vita Zahnfabrik, Bad Sackingen, Germany). Thirty-six specimens of each of the 4 materials were prepared and further divided into four subgroups of 9 each. **Table 1** includes details of their chemical structure (composition), manufacturer, brand name as well as the batch numbers. The conditions in which the four testing materials were divided are as follows: in the first subgroup the specimens were maintained 7 days in a desiccator at room temperature; in the second subgroup they were stored in an incubator for seven days in distilled water at 37^{o}C. These two subgroups did not have any further treatment. The third subgroup included specimens that were submitted to a 60,000 thermo cycling process for 40 days; and the fourth subgroup was submitted to a 120,000 thermo cycling process for 80 days. The thermo cycling process was made with Thermo Haake (Thermo Haake, Karlsruhe, Germany), each cycle lasted for 52 seconds, as follows: the dwell time within each bath (55^{o} and 5^{o}C) was 20 seconds, and the transferring time between baths was 6 seconds.

Table 1 – Details of materials included in research.

Material	Composition	Manufacturer	Batch
Vitablocs Mark II	Fine-particle feldspar ceramic	Vita Zahnfabrik	39350
IPS e.max CAD	Lithium disilicate glass ceramic	Ivoclar-Vivadent	78978
Lava Ultimate	Composite material silica and zirconia nanoparticles	3M ESPE	N674359
Vita Enamic	Polymer-infiltrated-ceramic-network 86% feldspathic ceramic	Vita Zahnfabrik	47791

The specimens were prepared for a three-point bending test to determine flexural strength, which is defined as the stress in a material at the moment before failure, flexural modulus, defined as the ratio of stress to strain, and how much energy the material is able to hold before bending. The CAD/CAM blocks were sectioned and prepared as beam specimens. First the materials were sectioned into slices out from the blocks, with each slice having 14.8mm x 12mm x 3mm (L x W x H) and then cut into beam shaped specimens measuring 14.8mm x 3.5mm x 3mm (L x W x H). They were cut with a precision diamond-saw blade mounted on a cutting machine (Isomet 1000; Buehler, Lake Bluff, USA). To avoid ceramic chipping during the cutting procedure and to achieve a more precise dimension, specimens were submitted to finishing and polishing procedures (Ecomet 6, Buehler, Lake Bluff, IL, USA) using the following sequence of sandpapers: 240, 400, 600, 800 and 1200 grit. Final dimensions (14mm x 3mm x 2.5mm) were confirmed by a digital caliper (Mitutoyo America Corporation, Illinois, USA).

The specimens were positioned on a three point-bending machine (Test Resources, Shakopee, MN, USA), and the tests were conducted at room temperature (23^{o}C) until the complete failure of the specimens. The maximum load was recorded and the following equations were used to calculate the flexural strength (σ) as the flexural modulus (E), respectively:

(1) $\sigma=3PL/bh^2$ – flexural strength

(2) $E=PL^3/bdh^3$ – flexural modulus

In both formulas P represents the maximum load in Newton (N), L is the distance between supports (12mm), b is the specimen width, h the specimen thickness, and in equation (2) d is the deflection mean on the entire slope of the linear portion. In both formulas b and h were measured with the digital caliper immediately before the test and were recorded in millimeters.

After the specimens were tested, three of each group were randomly selected and analyzed in the scanning electron microscope (SEM – FEI Nova Nanolab 200, Hillsboro, OR, USA) for fractographic analysis irrespective of different material behavior and the conditions applied. The values obtained after the three point-bending tests were submitted to statistical analysis, first Shapiro-Wilk and Levene’s tests were applied to make sure the data have the basic principles of normality distribution as well as homoscedasticity data. Subsequently, a two-way ANOVA was applied to identify possible statistical significant differences, and the Tukey’s test showed where these differences occurred.

Results

The mean (standard deviation) of flexural strength and flexural modulus are presented in **Table 2** for each ceramic individually. A two-way ANOVA **(Table A7)** and the *post-hoc* Tukey's test were performed for both variables. It was found statistical significant difference ($p<0.05$) among ceramics for both variables. IPS e.max CAD presented the highest value for flexural strength with a 396.19 MPa followed by Enamic with 153.22 MPa and Lava Ultimate with 149.78 MPa. The last two materials were not considered statistically different; however, Vitablocs Mark II presented the lowest value for flexural strength with 125.18 MPa. The results of flexural modulus presented statistical significant difference ($p<0.05$) for each material.

Table 2 – Mean and standard deviation of flexural strength (MPa) and flexural modulus (GPa) among the materials tested.

	Vitablocs	**IPS e.max CAD**	**Lava Ultimate**	**Enamic**
Flexural Strength	125.18 (10.07)[a]	396.19 (75.65)[b]	149.78 (28.99)[c]	153.22 (17.24)[c]
Flexural Modulus	49.92 (15.57)[a]	84.97 (11.12)[b]	12.90 (3.73)[c]	28.01 (5.99)[d]

[*]For each mechanical property individually, values denoted with same letters are not significantly ($p>0.05$).

Table 3 presents the manufacturer technical data for each material tested for both variables as well as the data from our research. For flexural strength, Vitablocs Mark II did not show statistical significant difference ($p<0.05$) within the conditions applied. IPS e.max CAD presented an increase on flexural strength after 7 days of water storage, which is considerably statistically different from the dry condition. Although after a long thermo cycling process, no decrease in performance was shown as expected. Lava Ultimate represents the class of resin nano ceramic materials. However, after 7 days of water storage and a long thermo cycling process, a decrease on flexural strength was shown. Considering the initial dry condition all other three were statistically different ($p<0.05$) for Lava Ultimate. Enamic classified as polymer-infiltrated-ceramic-network had a similar behavior to ceramic materials and did not present any statistical difference among the conditions to which it was submitted.

Table 3 – Flexural strength (MPa) and flexural modulus (GPa) of tested specimens submitted to different conditions and data from manufacturer's technical sheet.

	Condition	**Vitablocs**	**IPS e.max CAD**	**Lava Ultimate**	**Enamic**
Flexural Strength	Manufacturer	154	360	205	150-160
Flexural Modulus	Manufacturer	45	95	12	30
Flexural Strength	Desiccator	120.90^{a} (8.95)	304.94^{a} (32.29)	189.45^{a} (9.31)	164.08^{a} (13.57)
	Water Storage	132.65^{a} (9.03)	448.85^{b} (38.12)	146.33^{b} (23.04)	168.53^{a} (11.51)
	Thermo 60.000	122.19^{a} (8.26)	401.76^{c} (67.41)	135.55^{b} (18.23)	140.99^{a} (7.85)
	Thermo 120.000	124.99^{a} (10.95)	$429.22^{b,c}$ (106.32)	127.78^{b} (13.06)	139.30^{a} (11.80)
Flexural Modulus	Desiccator	40.70^{a} (16.12)	80.81^{a} (5.82)	12.72^{a} (3.71)	26.34^{a} (5.61)
	Water Storage	53.30^{b} (14.43)	93.08^{b} (3.20)	14.33^{a} (4.87)	29.15^{a} (2.04)
	Thermo 60.000	51.76^{b} (15.68)	78.64^{a} (7.44)	11.85^{a} (1.47)	29.16^{a} (4.66)
	Thermo 120.000	53.91^{b} (14.72)	$87.36^{a,b}$ (17.24)	12.69^{a} (4.19)	27.39^{a} (9.66)

[*]For each material individually as well as both mechanical properties, values denoted with same letters are not significantly (p>0.05).

Table 3 also presents the mean and standard deviation values for flexural modulus among the same conditions did for flexural strength. Vitablocs Mark II has shown a better performance after being hydrated, and the three conditions, in which the material was in contact with water, were not statistically different (p<0.05) from each other. However, they were different from the dry condition. The analysis of the behavior of IPS e.max CAD on flexural modulus was very similar to the one of flexural strength, even though there was a decrease after the long thermo cycling process, and it was considered with statistical significant difference (p<0.05) from the 7 day water storage. Lava Ultimate and Enamic were not statistically significant different (p<0.05) among the conditions tested.

In order to determine the fracture path of the tested materials figures 1 to 4 show the four materials in two different magnifications. The figure descriptions explain the different fracture characteristics of the materials. Some unexpected behaviors among the applied conditions were identified on the materials' surface. A common characteristic across the materials was that fracture surfaces were considered rough.

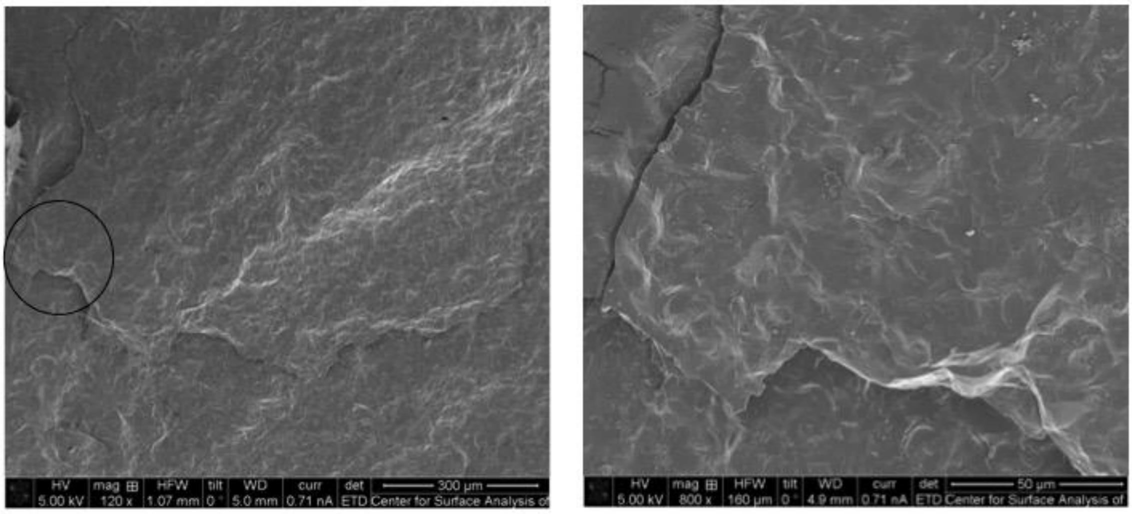

Figure 1 – Vitablocs Mark II – categorized as feldspathic porcelain, in the 120x magnification shows an appearance known as fish scale in the direction of the fracture, identified by the white arrow. A brittle fracture that occurred by cleavage is apparent over the surface in the two different magnifications, also a conchoidal fracture is visible as a result of breakage surface that appears like a wavelet.

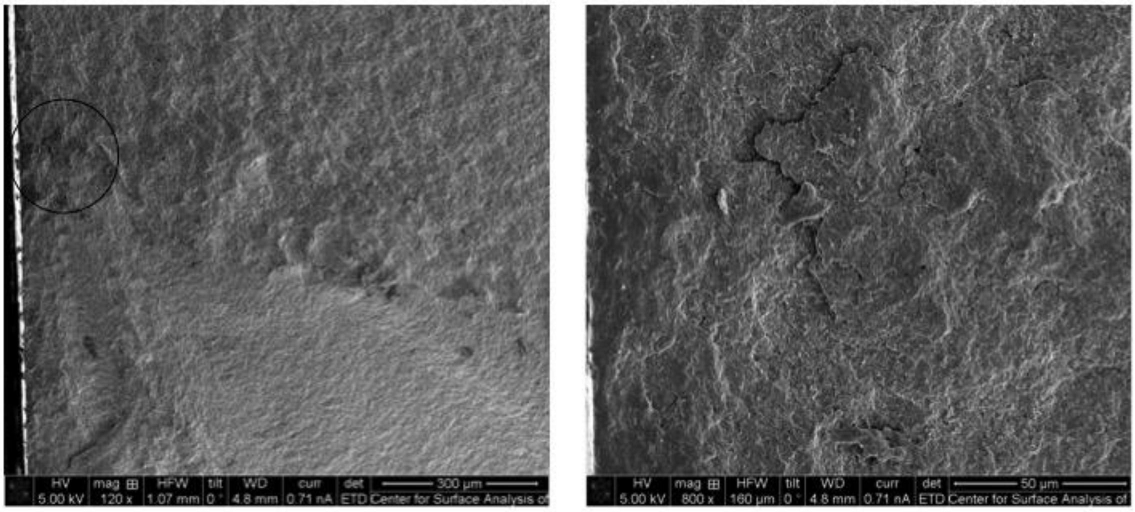

Figure 2 – IPS e.max CAD – classified as lithium disilicate glass ceramic, shows peaks and valleys in the 120x magnification due to the action of randomly oriented needle-like crystals.

The crystals cause the deflection of cracks propagation providing a substantial increase in the flexural strength. A brittle fracture is clear on the ceramic surface and flaws are also apparent. A porous region characterizes the center of the circle shown in the 800x magnification.

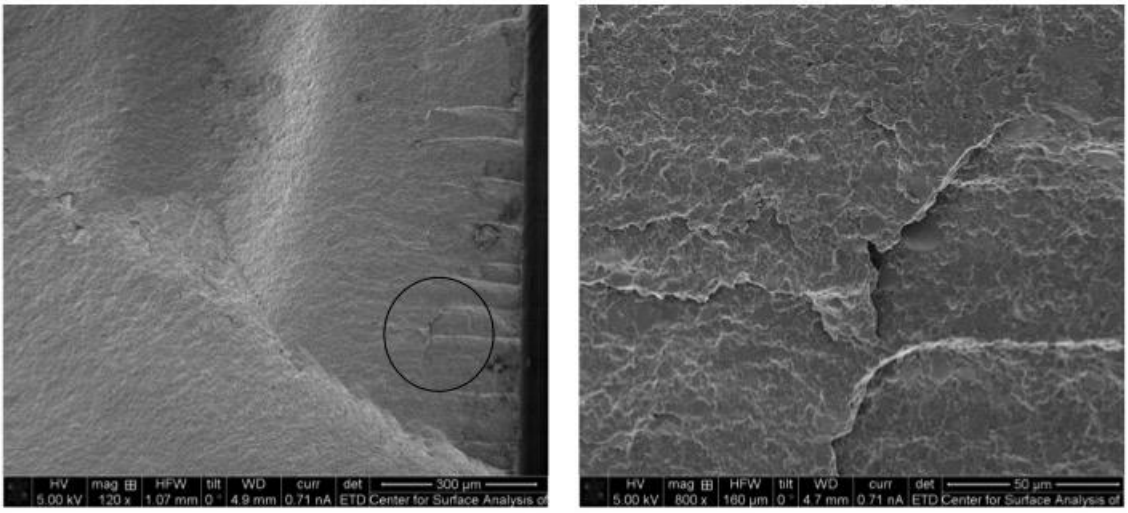

Figure 3 – Lava Ultimate – the resin nano ceramic material shows ductile fracture characteristics, in the 120x magnification can be identified on the right side of the figure by the undulation. At closer inspection (800x magnification) some small undulations and flaws are also visible characterizing the material with plastic behavior.

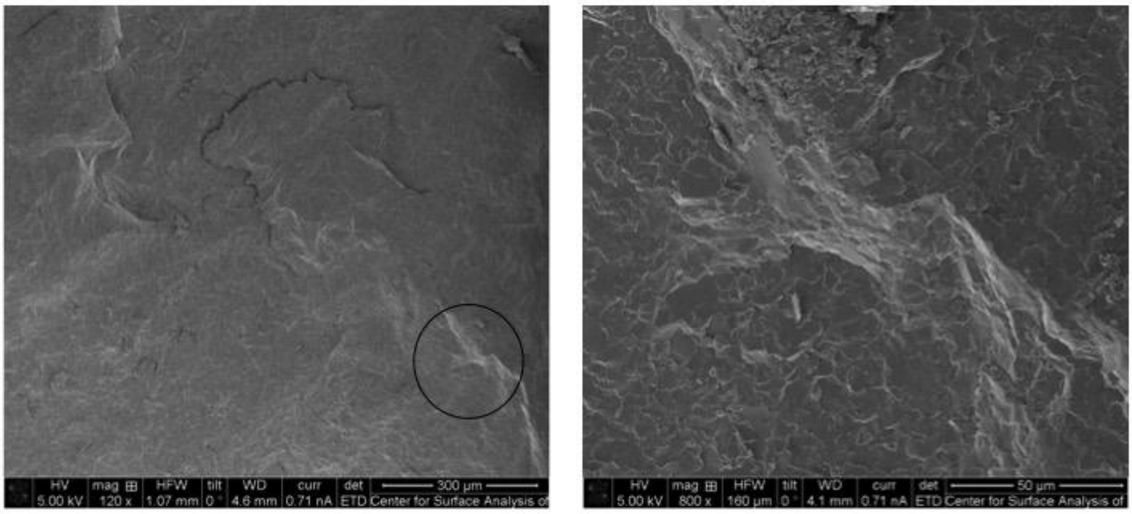

Figure 4 – Enamic – this polymer-infiltrated-ceramic-network, despite its polymer composition, the fracture characteristics it presents indicates a material with ceramic behavior. At 120x magnification the fish scale feature is clearly identified as is for feldspathic ceramic. Furthermore, at 800x magnification the ceramic network is visible and no plastic deformations were found. On the surface of both magnifications some porous region and also handling damage are apparent.

Discussion

This laboratory research had the aim of answering clinical questions related to the recent CAD/CAM materials mechanical properties such as elasticity modulus, flexural strength, and also fractographic features. The research and development involving the recent new resin nano ceramic and hybrid ceramic materials available in the market have raised important questions related to materials performance in comparison with well-known ceramic materials. The null hypothesis was that there is no difference on flexural strength and elasticity modulus before and after the long thermo cycling process for all materials within this research.

The mean for flexural strength and flexural modulus is presented in **Table 1** without the influence of thermo cycling. The results presented by each material have showed some interesting differences. The IPS e.max CAD shows the highest value for each one of the mechanical properties studied, besides being a monolithic glass ceramic, it is superior in deflection compared to the materials involved within this laboratory study. Similar studies[19] related to different preparation types showed the IPS e.max Press, which has exactly the same composition of IPS e.max CAD, with almost the same fracture resistance as yttrium stabilized zirconia, considered the most resistant ceramic in the dental market. Vitablocs Mark II the first glass ceramic used in the CEREC system presented the lowest value of flexural strength among the others, porcelain materials tend to be brittle and less flexible resulting in inferior deformation ability to absorb stress under high loading[6,20]. Nevertheless, the flexural modulus was just above IPS e.max CAD and did not change after a long thermo cycling test; this explains the material stability under clinical conditions[4,21,22].

Table 2 shows the flexural modulus given by manufacturers, and also the results by the thermo cycling process. Due to the resin component, Lava Ultimate has a low flexural modulus compared to the others. However, it did not change significantly after the thermo cycling. The flexural strength decreased after the conditions tested, as also indicated by other *in vitro* studies, which might occur due to the water absorption that the materials may suffer[10,11]. The only real hybrid ceramic available in the market is Vita Enamic, indicated for crowns, onlays/inlays, and veneers[3], has a major leucite-based phase infiltrated by a polymer-based network. This material presented a much higher flexural modulus in comparison to composite resin-based materials, despite the fact that it has a similar flexural strength. Furthermore, the values of flexural strength and flexural modulus did not change significantly after the long thermo cycling process, which makes the material similar to ceramics[23,24,25].

All SEM fractographies without exception demonstrated zones of roughness along the surface of all materials. None of the materials tested presented differences on the surface features across the different situations that they were submitted to. Vitablocs Mark II was the material with more cracks and hackles all over the surface characterizing brittle fracture. The IPS e.max CAD presented a rough surface with peaks and valleys, with small areas with cracks and hackles, a flaw from left to right, and in the middle of the picture a coarse hackle showing the crack path. Lava Ultimate displayed a clear ductile fracture identified by flaws with mixed attributes, porous seams, voids, and a coarse hackle. Although it is a composite resin-based material, the roughness of the surface is the same presented in all the ceramic materials.

The polymer-infiltrated-ceramic-network material demonstrated a mix of features that identifies hybrid characteristics; in the upper left corner similar flaws with mixed attributes present in Lava Ultimate are also apparent is this fractography. In the center and also the lower right corner, flaws and hackles characterizing a brittle fracture is also shown. Similar to what is observed in real clinical situations[26] the crack formation started in a zone of heavy loading, so the crack propagated along the material with characteristics typical of the materials compositions. Even though there are some laboratory studies in the literature, clinical research is necessary to better understand CAD/CAM materials behavior. It is important to understand that among factors influencing the longevity of all-ceramic restorations[27] correct occlusion and the polishing process will have a direct action on ceramic crowns, and might prevent small and incipient cracks or hackles. However, it was mentioned that the crack initiation started at the cement interface[27,28,29].

Conclusions

CAD/CAM materials mechanical properties have raised what the ceramic industry has of more value, knowledge. Furthermore, the selection criteria of CAD/CAM ceramics to do final restorations clearly will depend on the indication and the patient’s need. However, concerns about the performance of certain materials will always exist, research and development of CAD/CAM technology and materials will keep the valuable and important information accessible to clinicians and also the dentistry industry.

Within the limits of this investigation, it is concluded:

- Hybrid materials have a similar behavior to CAD/CAM ceramics in terms of flexural strength and may not suffer from opening marginal gaps within a great period of time.
- CAD/CAM ceramics are not influenced by the aging conditions and will perform better after being hydrated.
- CAD/CAM composite materials have a similar behavior to direct composites in terms of water absorption and decrease of flexural strength.

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4 CONSIDERAÇÕES FINAIS

A evolução tecnológica associada ao desenvolvimento dos materiais odontológicos através do aumento das pesquisas científicas, abriu um novo leque de discussão, além de novos horizontes a serem estudados. Conceitos básicos e fundamentados na literatura voltam a ser necessários ao clinico, bem como o pesquisador interessado em aprofundar-se no assunto.

O profissional que almeja trabalhar com a tecnologia CAD/CAM necessita de um conhecimento teórico e científico do comportamento atual das novas cerâmicas disponíveis no mercado para serem utilizadas no atual sistema. Princípios muitas vezes negligenciados ou até mesmo desconhecidos pelo clinico; como índice de fragilidade, também conhecido como tenacidade à fratura das cerâmicas dentais, englobam os conceitos atuais e anteriormente utilizados, necessários a este novo desafio.

Obviamente a tecnologia CAD/CAM trouxe muitos avanços, como: diminuir os custos de fabricação de coroas protéticas, otimizar o atendimento clinico, aumentar a precisão das peças, entre muitos outros. Porém, nossos resultados mostraram que a fragilidade das cerâmicas dentais está diretamente relacionada a sua dureza, evidenciando que quanto mais duro o material, inferior deve ser seu desempenho do ponto de vista da usinabilidade. Entretanto, a escolha do material a ser utilizado pelo clinico para a finalização de seu trabalho protético deve estar diretamente relacionado as limitações impostas pelos novos equipamentos. Por outro lado permite ao clinico escolher diretamente qual material utilizar exigindo o conhecimento relacionado as propriedades mecânicas dos materiais disponíveis.

A correlação de *Pearson* mostrou que o indice de fragilidade está diretamente relacionado com a dureza Vickers, quanto maior sua dureza mais frágil será o material. Quando nos referenciamos a tecnologia CAD/CAM, que necessita de uma fresadora para confecção das coroas protéticas, é importante entendermos limitações tais como; espessura do material que será um fator de complicação durante a fresagem. Situações clinicas limitantes ou que exijam a necessidade de preparos conservadores podem impor desafios, que devem ser contornados pela escolha correta do material, ou ainda a utilização de outros meios para a realização do trabalho protético.

Entretanto os novos materiais disponíveis, devido a fabricação industrial das cerâmicas, sem necessidade que alterem seu estado para o processo de confecção das coroas protéticos novas possibilidades tornaram-se possíveis para a industria. Os materiais apresentados nos nossos estudos conhecidos por resina nano cerâmica (Lava Ultimate) e cerâmica híbrida (Vita Enamic) mostraram possuir excelentes propriedades mecânicas, permitindo em muitas ocasiões substituirem as já conhecidas cerâmicas vítreas.

A despeito das propriedades individuais de cada material existem algumas particularidades que são importantes salientar. A cerâmica de disilicato de lítio fabricada pela Ivoclar – Vivadent apesar de possuir o mais alto valor do índice de fragilidade entre os materiais testados, consequentemente a pior performance nesse quesito em sua forma pré-sinterizada, mostrou-se o material com a maior resistência à fratura, modulo de elasticidade e resistência à flexão após o processo de cristalização, não alterando suas propriedades após ter sido submetida a um longo processo de termociclagem. A cerâmica feldspática conhecida por Vitablocs Mark II, a primeira a ser utilizada com a tecnologia CAD/CAM veio sofrendo modificações em sua composição ao longo dos anos. Assim como o IPS e.max CAD teve um índice de fragilidade similar e não apresentou alterações em suas propriedades mecânicas após o processo de termociclagem.

Os materiais desenvolvidos recentemente incluídos em nossa pesquisa (Lava Ultimate / Vita Enamic), como anteriormente salientado mostraram possuir excelentes propriedades mecânicas, com um indice de fragilidade de valor inferior a todos os outros no caso do Lava Ultimate, sugerindo assim uma maior capacidade de usinabilidade por parte deste material. Entretanto, ambos materiais após o periodo de termociclagem apresentaram valores menores do que os inicias de resistência à fratura, modulo de elasticidade e resistência à flexão, evidenciando que sofram absorção de água quando em contato com meios úmidos. Os valores finais apresentaram similaridades a cerâmica feldspática que desde o início performou com valores inferiores.

Todos os materiais testados apresentaram resultados dentro do esperado e devem ser criticamente estudados em situações in vivo para que situações reais também possam servir de embasamento para as mais diversas condições clinicas. Além do mais, diante dos resultados encontrados na literatura, as cerâmicas vítreas, e materiais híbridos que apresentam formulações variadas possuem excelentes propriedades mecânicas, capacidade estética; e através do sistema CAD/CAM redução de custos e agilidade nas consultas clinicas, porém as mais diversas situações clinicas devem ser criticamente avaliadas pelo profissional em questão e comparadas com os sistemas tradicionais a fim de que as vantagens sejam realmente evidenciadas.

É importante reafirmar que não somente estudos clinicos, mas pesquisas laboratorias envolvendo as cerâmicas disponíveis no sistema CAD/CAM fazem-se necessárias para avaliação de suas respectivas propriedades, fornecendo assim informações científicas aos clinicos para a obtenção dos melhores resultados.

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APÊNDICE A

Tabela A1 – Teste para normalidade Shapiro-Wilk.

		Kolmogorov-Smirnov[a]			Shapiro-Wilk		
	ceramic	Statistic	df	Sig.	Statistic	df	Sig.
hardness	empress	.160	13	.200*	.909	13	.175
	emax	.159	13	.200*	.944	13	.517
	cerec	.150	13	.200*	.943	13	.503
	lava	.206	13	.136	.864	13	.043
brittle	empress	.172	13	.200*	.950	13	.598
	emax	.110	13	.200*	.965	13	.822
	cerec	.138	13	.200*	.944	13	.517
	lava	.227	13	.065	.897	13	.123

Fonte: Elaboração própria

Tabela A2 – Teste Levene para homogeneidade de variâncias.

	Levene Statistic	df1	df2	Sig.
hardness	4.623	3	48	.006
brittle	6.614	3	48	.001

Fonte: Elaboração própria

Tabela A3 – Análise de variância.

		Sum of Squares	df	Mean Square	F	Sig.
hardness	Between Groups	2810019.103	3	936673.034	3175.371	.000
	Within Groups	14159.070	48	294.981		
	Total	2824178.173	51			
brittle	Between Groups	20.385	3	6.795	492.903	.000
	Within Groups	.662	48	.014		
	Total	21.047	51			

Fonte: Elaboração própria

Tabela A4 – Teste *post-hoc* Games-Howell.

Dependent Variable		(I) ceramic	(J) ceramic	Mean Difference (I-J)	Std. Error	Sig.	99,9% Confidence Interval Lower Bound	99,9% Confidence Interval Upper Bound
hardness	Games-Howell	empress	emax	-111.88308*	7.39443	.000	-132.3625	-91.4037
			cerec	-23.45923*	8.15865	.039	-45.9660	-.9524
			lava	483.01846*	6.08250	.000	465.3858	500.6511
		emax	empress	111.88308*	7.39443	.000	91.4037	132.3625
			cerec	88.42385*	7.33256	.000	68.1245	108.7232
			lava	594.90154*	4.91933	.000	580.7922	609.0108
		cerec	empress	23.45923*	8.15865	.039	.9524	45.9660
			emax	-88.42385*	7.33256	.000	-108.7232	-68.1245
			lava	506.47769*	6.00714	.000	489.0729	523.8825
		lava	empress	-483.01846*	6.08250	.000	-500.6511	-465.3858
			emax	-594.90154*	4.91933	.000	-609.0108	-580.7922
			cerec	-506.47769*	6.00714	.000	-523.8825	-489.0729
brittle	Games-Howell	empress	emax	-.75154*	.05709	.000	-.9107	-.5924
			cerec	-.53385*	.04344	.000	-.6537	-.4140
			lava	.87231*	.03333	.000	.7758	.9688
		emax	empress	.75154*	.05709	.000	.5924	.9107
			cerec	.21769*	.05596	.005	.0612	.3742
			lava	1.62385*	.04852	.000	1.4816	1.7661
		cerec	empress	.53385*	.04344	.000	.4140	.6537
			emax	-.21769*	.05596	.005	-.3742	-.0612
			lava	1.40615*	.03135	.000	1.3156	1.4967
		lava	empress	-.87231*	.03333	.000	-.9688	-.7758
			emax	-1.62385*	.04852	.000	-1.7661	-1.4816
			cerec	-1.40615*	.03135	.000	-1.4967	-1.3156

Fonte: Elaboração própria

Tabela A5 – Two-way ANOVA resistência à fratura.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.	Partial Eta Squared
Corrected Model	2.175[a]	15	.145	40.551	.000	.826
Intercept	37.767	1	37.767	10560.384	.000	.988
ceramic	1.925	3	.642	179.423	.000	.808
condition	.048	3	.016	4.479	.005	.095
ceramic * condition	.202	9	.022	6.285	.000	.306
Error	.458	128	.004			
Total	40.400	144				
Corrected Total	2.633	143				

Fonte: Elaboração própria

Tabela A6 – Tukey *post-hoc* teste (p>0,05) comparação de pares (continua).

ceramic	(I) condition	(J) condition	Mean Difference (I-J)	Std. Error	Sig.[b]	95% Confidence Interval for Difference[b] Lower Bound	Upper Bound
vita	dry sample	water storage	-.027	.028	.334	-.083	.028
		thermo60	-.031	.028	.267	-.087	.024
		thermo120	-.084*	.028	.004	-.140	-.028
	water storage	dry sample	.027	.028	.334	-.028	.083
		thermo60	-.004	.028	.884	-.060	.052
		thermo120	-.057*	.028	.047	-.112	-.001
	thermo60	dry sample	.031	.028	.267	-.024	.087
		water storage	.004	.028	.884	-.052	.060
		thermo120	-.052	.028	.065	-.108	.003
	thermo120	dry sample	.084*	.028	.004	.028	.140
		water storage	.057*	.028	.047	.001	.112
		thermo60	.052	.028	.065	-.003	.108
emax	dry sample	water storage	-.104*	.028	.000	-.160	-.049
		thermo60	-.081*	.028	.005	-.137	-.025
		thermo120	-.125*	.028	.000	-.181	-.070
	water storage	dry sample	.104*	.028	.000	.049	.160
		thermo60	.023	.028	.410	-.033	.079
		thermo120	-.021	.028	.458	-.077	.035
	thermo60	dry sample	.081*	.028	.005	.025	.137
		water storage	-.023	.028	.410	-.079	.033
		thermo120	-.044	.028	.119	-.100	.012
	thermo120	dry sample	.125*	.028	.000	.070	.181

Tabela A6 – Tukey *post-hoc* teste (p>0,05) comparação de pares (conclusão).

		water storage	.021	.028	.458	-.035	.077
		thermo60	.044	.028	.119	-.012	.100
lava	dry sample	water storage	-.013	.028	.642	-.069	.043
		thermo60	.090*	.028	.002	.034	.146
		thermo120	.099*	.028	.001	.043	.155
	water storage	dry sample	.013	.028	.642	-.043	.069
		thermo60	.103*	.028	.000	.048	.159
		thermo120	.112*	.028	.000	.056	.168
	thermo60	dry sample	-.090*	.028	.002	-.146	-.034
		water storage	-.103*	.028	.000	-.159	-.048
		thermo120	.009	.028	.763	-.047	.064
	thermo120	dry sample	-.099*	.028	.001	-.155	-.043
		water storage	-.112*	.028	.000	-.168	-.056
		thermo60	-.009	.028	.763	-.064	.047
enamic	dry sample	water storage	-.033	.028	.243	-.089	.023
		thermo60	.024	.028	.399	-.032	.080
		thermo120	.062*	.028	.031	.006	.117
	water storage	dry sample	.033	.028	.243	-.023	.089
		thermo60	.057*	.028	.046	.001	.113
		thermo120	.095*	.028	.001	.039	.150
	thermo60	dry sample	-.024	.028	.399	-.080	.032
		water storage	-.057*	.028	.046	-.113	-.001
		thermo120	.038	.028	.182	-.018	.094
	thermo120	dry sample	-.062*	.028	.031	-.117	-.006
		water storage	-.095*	.028	.001	-.150	-.039
		thermo60	-.038	.028	.182	-.094	.018

Fonte: Elaboração própria

Tabela A7 – Two-way ANOVA resistência flexural.

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	1888873.374[a]	15	125924.892	99.076	.000
Intercept	6116648.166	1	6116648.166	4812.483	.000
ceramic	1751423.507	3	583807.836	459.331	.000
condition	17522.381	3	5840.794	4.595	.004
ceramic * condition	119927.486	9	13325.276	10.484	.000
Error	162687.519	128	1270.996		
Total	8168209.059	144			
Corrected Total	2051560.892	143			

Fonte: Elaboração própria