

UNIVERSIDADE FEDERAL DE PELOTAS
Programa de Pós-Graduação em Odontologia



Dissertação

Tetrahidrofurano como solvente de adesivos odontológicos

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**TETRAHIDROFURANO COMO SOLVENTE DE ADESIVOS
ODONTOLÓGICOS**

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PROJETO DE PESQUISA¹

¹ Projeto redigido segundo o Manual de Normas para Dissertações da Universidade Federal de Pelotas disponível em: <<http://www.ufpel.tche.br/prg/sisbi>>. Acesso em: 20 fev. 2009.

RESUMO

FONTES, Silvia Terra. **Tetrahidrofurano como solvente de adesivos odontológicos**. 2009. 77p. Dissertação de Mestrado – Programa de Pós-Graduação em Odontologia, Universidade Federal de Pelotas, Pelotas, RS, Brasil.

O objetivo deste estudo será avaliar o desempenho do tetrahidrofurano (THF) como solvente na composição do *primer* de um adesivo odontológico experimental através da resistência de união à microtração em dentina após 24h e 6 meses de armazenamento em água. Sessenta incisivos bovinos serão distribuídos aleatoriamente em dez grupos, conforme o tipo de solvente: 1) THF, 2) acetona, 3) etanol, 4) água, 5) THF/água, 6) acetona/água, 7) etanol/água, 8) All Bond 2 (acetona/etanol/água), 9) Opti Bond FL (etanol/água) e 10) Scotch Bond (água). O desgaste na face vestibular dos dentes será realizado até exposição da dentina superficial com lixas de carbeto de silício sob refrigeração. Depois que a superfície dentinária for condicionada com gel de ácido fosfórico a 35% (15s) e lavada com água (30s), o excesso de água superficial será removido com papel absorvente. Nos grupos experimentais, o *primer* será aplicado sob agitação constante (30s) seguido de jato de ar por (10s) a distância de 10cm, a fim de auxiliar a evaporação do solvente. Uma camada de resina adesiva experimental será aplicada e fotoativada (20s). Os sistemas adesivos de marcas comerciais serão aplicados conforme instruções dos fabricantes. As restaurações serão realizadas com compósito restaurador pela técnica incremental. Os incrementos serão fotoativados individualmente a $1400\text{mW}/\text{cm}^2$. Após o período de 24 horas de armazenamento em água a 37°C , as amostras serão seccionadas obtendo-se uma área de secção transversal de aproximadamente $0,5\text{mm}^2$. Metade dos corpos de prova será testada imediatamente e a outra metade será mantida sob as mesmas condições de armazenamento para avaliação após 6 meses. A resistência de união à microtração (MPa) será testada através de uma máquina universal de ensaios mecânicos ($1,0\text{mm}/\text{min}/100\text{N}$). As porções correspondentes à dentina serão analisadas por microscopia de luz (500X) e classificadas de acordo com o padrão de fratura em: CD) falhas coesivas em dentina, CC) falhas coesivas em compósito restaurador, A) falhas adesivas, M) falhas mistas ou FP) falhas prematuras. Significância estatística será considerada quando $\alpha=0.05$.

Palavras-chave: Adesivo. Dentina. Materiais dentários. Microtração. Resistência de união. Solvente.

ABSTRACT

FONTES, Silvia Terra. **Tetrahydrofuran as solvent in dental adhesive systems**. 2009. 77p. Dissertation (Master degree) – Post Graduate Program, School of Dentistry, Federal University of Pelotas, Pelotas, RS, Brazil.

The aim of this study is to evaluate the performance of tetrahydrofuran (THF) as solvent in the primer composition of an experimental adhesive system on the resin-dentin microtensile bond strength after 24h and 6 months of water storage. Sixty bovine incisors will be randomly separated into ten groups, according to the solvent type: 1) THF, 2) acetone, 3) ethanol, 4) water, 5) THF/water, 6) acetone/water, 7) ethanol/water, 8) All Bond 2 (acetone/ethanol/water), 9) Opti Bond FL (ethanol/water) e 10) Scotch Bond (water). Vestibular enamel will be removed to expose the superficial dentin and further polished with grit silicon-carbide under irrigation. After acid etching with 35% phosphoric acid (15s), the surfaces will be rinsed with water (30s) and the excess of superficial water will be removed with absorbent paper. Each experimental primer will be applied with agitation (30s) followed by a gently air stream (10s) at a distance of 10cm to help solvent evaporation. One coat of an experimental adhesive resin will be applied and light-activated (20s). The marketed available adhesive systems will be used according manufacturer instructions. Resin composite restorations will be constructed incrementally. Each layer of composite will be light-activated individually at $1400\text{mW}/\text{cm}^2$. After water storage for 24h at 37°C , the restored teeth will be sectioned to obtain sticks with a cross-sectional area of approximately 0.5mm^2 . Half of specimens will be tested after 24 hours and the other half will be maintained in the same storage conditions to be evaluated after 6 months. Microtensile bond strength (MPa) will be evaluated with an universal testing machine (1.0mm/min/100N). The half corresponding to dentin will be evaluated in a light microscope (500X) and classified as: CD) cohesive in dentin, CC) cohesive in resin composite, A) adhesive, M) mixed or PD) premature debonded. Statistical significance will be set at $\alpha=0.05$.

Keywords: Adhesive. Bond strength. Dental materials. Dentin. Microtensile. Solvent.

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1 INTRODUÇÃO

Atualmente, os sistemas adesivos convencionais com três etapas de aplicação apresentam desempenho mais favorável a longo prazo do que os sistemas adesivos de técnica simplificada (BRESCHI et al., 2008, DE MUNCK et al., 2003, OSORIO et al., 2008). Entretanto, os adesivos convencionais apresentam etapas críticas de aplicação, que consistem no manejo clínico de uma umidade de superfície ideal (MANSO et al., 2008, REIS et al., 2004) e de completa evaporação dos solventes (GIANNINI et al., 2008, IKEDA et al., 2005). Tanto a desidratação do substrato (MACIEL et al., 1996, NAKAJIMA et al., 2002), quanto a retenção de solventes orgânicos e água (YIU et al., 2005), afetam adversamente a infiltração e a polimerização dos monômeros resinosos.

Monômeros hidrófilos dissolvidos em solventes voláteis assumem papel imprescindível na hibridização dentinária, devido à natureza úmida do substrato (TAY; PASHLEY, 2003), e na reexpansão da rede de fibras de colágeno colapsadas em decorrência de secagem excessiva (AGEE et al., 2006, GARCIA et al., 2005, PASHLEY et al., 2003). Além disso, os solventes facilitam a evaporação da água residual, que deve ser completamente eliminada antes da polimerização (DICKENS; CHO, 2005, GIANNINI et al., 2008, HOLMES et al., 2007). Devido ao baixo peso molecular e à alta capacidade de difusão dos solventes, tais componentes atuam positivamente à medida que reduzem a viscosidade e aumentam o potencial hidrofílico da solução (CHO; DICKENS, 2004, GARCIA et al., 2005, VAN LANDUYT et al., 2007). Por estas razões, considerável número de produtos disponíveis comercialmente tem utilizado etanol, acetona e/ou água como veículos aos monômeros resinosos, tornando-se elementos indispensáveis às formulações (ABATE et al., 2000, VAN LANDUYT et al., 2007).

No entanto, para conferir maior durabilidade clínica, os sistemas adesivos deveriam apresentar formulações à base de monômeros hidrófobos, evitando a presença de monômeros hidrófilos, solventes orgânicos e água em sua composição (BRESCHI et al., 2008, TAY; PASHLEY, 2003, TAY et al., 2007). Estes

componentes estão relacionados a uma série de implicações clínicas, acarretando uma maior suscetibilidade à sorção de água, solubilidade e degradação da interface de união (CARRILHO et al., 2005, DE MUNCK et al., 2003, MALACARNE et al., 2006, YIU et al., 2006). Ademais, os solventes usados em Odontologia devem atender aos requisitos de biocompatibilidade e baixo custo (VAN LANDUYT et al., 2007).

A habilidade de reexpandir a rede de fibrilas de colágeno colapsadas, em decorrência da desidratação, está relacionada com a capacidade dos solventes em formar ligações de hidrogênio (AGEE et al., 2006, GARCIA et al., 2005, PASHLEY et al., 2003), enquanto a taxa de evaporação é dependente da temperatura de ebulição e vapor de pressão desses componentes (ABATE et al., 2000, IKEDA et al., 2005, REIS et al., 2008). A água, por exemplo, é um solvente inorgânico, altamente polar e amplamente utilizado nas formulações (VAN LANDUYT et al., 2007). Através de sua habilidade para prevenir ou romper interações intermoleculares é capaz de reverter situações de colapamento das fibrilas de colágeno (NAKAJIMA et al., 2002, PASHLEY et al., 2003). No entanto, sua alta temperatura de ebulição e baixo valor de vapor de pressão podem predizer sua maior dificuldade de volatilização em comparação com outros solventes (ABATE et al., 2000, VAN LANDUYT et al., 2007). Isto significa que mesmo após os eventos de evaporação, solventes orgânicos e água podem permanecer retidos, inclusive se houver formação de ligações de hidrogênio entre solventes e monômeros hidrófilos, favorecendo ainda mais sua retenção (IKEDA et al., 2005, YIU et al., 2005). Desse modo, solventes e água residuais podem ser considerados fatores predisponentes à degradação (BRESCHI et al., 2008, DE MUNCK et al., 2003).

Na tentativa de solucionar inconvenientes desta natureza, uma determinada empresa de produtos odontológicos incorporou, recentemente, terc-butanol como solvente à formulação de um sistema adesivo. A adição deste componente foi justificada pela sua estabilidade química e capacidade de evaporação (VAN LANDUYT et al., 2007). Estudos prévios testaram a aplicabilidade do metanol, verificando seu comportamento favorável como solvente, devido a sua habilidade de evaporação e reexpansão da matriz desmineralizada (CARVALHO et al., 2003, PASHLEY et al., 2003). Apesar disso, o metanol não tem sido utilizado como solvente na composição dos adesivos atualmente disponíveis.

Dentro deste contexto, é evidente que ainda existe necessidade de se buscar novas possibilidades como solventes para uso em Odontologia, a fim de otimizar o desempenho dos sistemas adesivos atuais. O tetrahidrofurano (THF) consiste em um composto orgânico heterocíclico (fig. 1) com capacidade de evaporação semelhante à acetona. Trata-se de um solvente polar aprótico capaz de dissolver componentes polares e apolares (SOLOMONS; FRYHLE, 2001). Apesar do THF apresentar características que se assemelham às propriedades dos solventes tradicionalmente empregados nas formulações (tab. 1), são poucas as informações encontradas acerca de seu comportamento em materiais odontológicos (BRAUER; TERMINI, 1972).

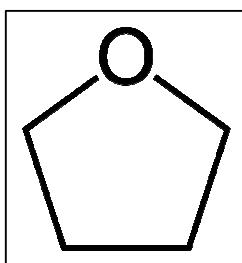


Figura 1 - Fórmula estrutural do THF.

Tabela 1 - Propriedades físicas de alguns solventes

Nome	Peso molecular (g/mol)	Ponto de ebulição (°C)	Vapor de pressão a 20°C (mmHg)
THF	72,1	65,4	173
Acetona	58,0	56,2	184
Etanol	46,0	78,5	43,9
Água	18,0	100	17,5

Fonte: SOLOMONS; FRYHLE, 2001.

2 JUSTIFICATIVA

Frente ao papel imprescindível desempenhado pela água e solventes orgânicos incorporados aos sistemas adesivos, o tetrahidrofurano (THF) pode surgir como um solvente alternativo viável para utilização em Odontologia. Tal composto apresenta propriedades físico-químicas que favorecem sua indicação como solvente na formulação de sistemas adesivos. Ademais, seu desempenho satisfatório poderá contribuir para a evolução e o desenvolvimento de novos biomateriais para aplicação odontológica.

3 OBJETIVO

O objetivo do presente estudo será investigar o desempenho do THF como solvente orgânico na composição do *primer* de um sistema adesivo experimental através de teste de resistência de união à microtração em dentina após 24 horas e 6 meses de armazenamento em água.

4 HIPÓTESE NULA

A hipótese nula a ser testada é que a variação do tipo de solvente utilizado na composição do *primer* de sistemas adesivos experimentais não terá influência nos valores de resistência de união à microtração imediata e a longo prazo.

5 MATERIAIS E MÉTODOS

5.1 Considerações iniciais

Este projeto de pesquisa será submetido ao Conselho Coordenador de Ensino, Pesquisa e Extensão (COCEPE) da Universidade Federal de Pelotas (UFPel) para processo de aprovação.

5.2 Formulação dos *primers* e da resina adesiva experimental

Os reagentes usados neste estudo serão obtidos diretamente dos fabricantes, sem receber tratamentos adicionais. As proporções em massa dos produtos experimentais serão realizadas em balança analítica de precisão (AG 200, Gehaka, Brasil), isotermicamente, a temperatura de 23°C (± 1) e umidade relativa de 70% (± 5).

Sete *primers* dentinários serão preparados a partir de quantidades fixas de 50% de HEMA (2-hidroxietil metacrilato) (Aldrich, USA), 10% de monômero ácido (LIMA GDA et al., 2008) e 40% de solvente (Labsynth, Brasil). Estas concentrações permanecerão constantes para todos os grupos, variando somente o tipo de solvente que será utilizado na forma pura e em combinação com água (tab. 2).

Tabela 2 - Composição (% em massa) dos sete *primers* experimentais

Reagentes	HEMA	Monômero	THF	Acetona	Etanol	Água	Total
Grupo	ácido						
1	50	10	40	0	0	0	100
2	50	10	0	40	0	0	100
3	50	10	0	0	40	0	100
4	50	10	0	0	0	40	100
5	50	10	20	0	0	20	100
6	50	10	0	20	0	20	100
7	50	10	0	0	20	20	100

HEMA: 2-hidroxietil metacrilato; THF: tetrahidrofurano.

Uma resina adesiva (CDC-Bio/UFPel, Brasil), formulada através da mistura intensa de bisfenol-A glicidil dimetacrilato (Bis-GMA), trietileno glicol dimetacrilato (TEGDMA), HEMA, iniciadores de polimerização e estabilizadores, será usada como adesivo de cobertura após aplicação do *primer* para todos os grupos experimentais.

Depois de prontas, todas as misturas serão homogeneizadas por 15 minutos em cuba ultrassônica (Plana, Brasil) e armazenadas, hermeticamente, até o momento de serem utilizadas.

5.3 Produtos de marcas comerciais (grupos controle)

Serão utilizados três adesivos disponíveis comercialmente, contendo diferentes tipos de solventes, os quais servirão como grupos controle aos sistemas adesivos experimentais. O nome comercial, os dados do fabricante e a composição dos produtos empregados neste estudo estão descritos na tab. 3.

Tabela 3 - Características dos adesivos de marcas comerciais

Grupo	Nome (fabricante)	Composição
8	All Bond 2 (Bisco, Schaumburg, IL, USA)	1) Ácido: H ₃ PO ₄ 35% 2) <i>Primer</i> A: NTG-GMA, acetona, etanol e água 3) <i>Primer</i> B: BPDM, fotoiniciador, acetona 4) Adesivo: Bis-GMA, HEMA, UDMA
9	Opti Bond FL (Kerr, Orange, CA, USA)	1) Ácido: H ₃ PO ₄ 35% 2) <i>Primer</i> : HEMA, GPDM, PAMA, CQ, BHT, etanol e água 3) Adesivo: Bis-GMA, HEMA, GDMA, CQ, EDMAB, partículas de sílica, bário, fluorsilicato de sódio, alumínio borosilicato de bário, fotoiniciador
10	Adper Scotch Bond (3M ESPE, St Paul, MN, USA)	1) Ácido: H ₃ PO ₄ 35% 2) <i>Primer</i> : HEMA, ácido polialcenóico, água 3) Adesivo: Bis-GMA, HEMA, amina terciária, fotoiniciador

BHT: 2,6-di-terc-butil-p-hidroxi tolueno; Bis-GMA: bisfenol-A glicidil dimetacrilato; BPDM: bisfenil dimetacrilato; CQ: canforoquinona; EDMAB: etil 4-dimetil amino benzoato; GDMA: glicerol dimetacrilato; GPDM: glicerofosfato dimetacrilato; HEMA: 2-hidroxietil metacrilato; H₃PO₄: ácido fosfórico; NTG-GMA: np-tolil glicidil metacrilato; PAMA: ácido monometacrilato phtalico; UDMA: uretano dimetacrilato.

5.4 Preparo dos dentes

Sessenta incisivos inferiores bovinos serão obtidos em frigorífico e imediatamente armazenados em solução desinfetante de cloramina-T 0,5% durante sete dias. Após o período de desinfecção, será removido o ligamento periodontal e o tecido pulpar dos dentes, enquanto a raiz será seccionada. Os dentes serão colocados em água destilada e acondicionados em *freezer*, aonde permanecerão congelados até o momento de serem utilizados.

Dentro do período de três meses a partir da data de extração, será escolhida aleatoriamente a quantidade de seis dentes para cada grupo em teste. A face lingual será planificada e a superfície vestibular será desgastada até exposição da dentina coronal superficial. A padronização da superfície dentinária será conseguida através do polimento das amostras por 60s, usando lixas impermeáveis de carbetto de silício (SiC) com granulação #600 acopladas a uma politriz universal (Aropol-E, Arotec, Brasil), usada em velocidade de 50rpm sob irrigação constante.

5.5 Procedimentos adesivos

Todos os procedimentos adesivos serão executados por um único operador previamente treinado.

Após aplicação de jato de ar, deixando a superfície visivelmente seca, o condicionamento de toda superfície de dentina será realizado com gel de ácido fosfórico 35% (CDC-Bio/UFPel, Brasil) por 15s. Depois desta etapa, será realizada lavagem abundante da superfície com água por 30s, seguida de remoção do excesso de umidade superficial com papel absorvente. De acordo com o grupo experimental, o *primer* será aplicado com auxílio de um pincel descartável sob agitação constante por 30s. Em seguida, será empregado jato seco de ar por 10s à distância de 10cm para auxiliar a evaporação do solvente. Uma fina camada do componente adesivo será aplicada uniforme e regularmente sobre toda a superfície de dentina e fotoativada por 20s com aparelho LED (Radii, SDI, Austrália) com intensidade de 1400mW/cm².

Nos grupos controle, os sistemas adesivos serão utilizados conforme recomendações dos fabricantes (tab. 4).

Tabela 4 - Instruções de uso dos adesivos de marcas comerciais

Grupo	Nome comercial	Modo de aplicação
8	All Bond 2	a, b, c ¹ , d ² , e, f, g ²
9	Opti Bond FL	a, b, c ² , d ¹ , e, f, g ³
10	Scotch Bond	a, b, c ² , d ¹ , e, f, g ¹

a. Condicionamento com ácido fosfórico 35% (15s); b. Enxágue abundante com água (15s); c¹. Jato de ar (1-2s); c². Jato de ar (5s); d¹. Aplicação de uma camada de *primer*; d². Mistura do *primer* A + *primer* B e aplicação de 5 camadas; e. Jato de ar (5s); f. Aplicação do adesivo; g¹. Fotoativação (10s); g². Fotoativação (20s); g³. Fotoativação (30s).

As restaurações serão confeccionadas com compósito restaurador (Charisma, Heraeus Kulzer, Alemanha), utilizando quatro incrementos de aproximadamente 1,0mm de espessura. Cada um dos incrementos será fotoativado individualmente por 20s.

Os dentes restaurados serão devidamente identificados por grupos e armazenados em água destilada no interior de estufa (Q316M2, Quimis, Brasil) a temperatura de 37°C, aonde permanecerão durante 24 horas.

5.6 Preparo dos corpos de prova e teste de resistência de união

Após o período de 24 horas, as amostras serão seccionadas perpendicularmente à interface adesiva em duas direções, utilizando disco de corte diamantado acoplado a cortadeira de precisão (Isomet 1000, Buehler, EUA) com velocidade de 400rpm.

Serão obtidos em média seis palitos por dente, sendo que os espécimes provenientes de uma mesma amostra serão distribuídos de forma aleatória em dois subgrupos (24 horas e 6 meses), contendo 18 unidades cada um. Desta forma, além da avaliação da resistência de união imediata, serão guardados corpos de prova para avaliação em testes de longevidade após 6 meses de armazenagem em água a 37°C. Os palitos terão área de secção transversal de aproximadamente 0,5mm², cujas dimensões serão aferidas com um paquímetro digital (500-144B, Mitutoyo, Brasil) com precisão de 0,01mm.

Os corpos de prova serão fixados individualmente em um dispositivo metálico desenvolvido para ensaios de microtração com auxílio de um adesivo instantâneo à base de cianoacrilato (Super Bonder Gel, Loctite, Brasil). O tracionamento será

realizado até fratura em uma máquina universal de ensaios mecânicos (DL 500, EMIC, Brasil), utilizando velocidade de 1,0mm/min e célula de carga de 100N.

5.7 Análise do modo de fratura

As porções fraturadas correspondentes à dentina serão analisadas através de microscopia de luz disponível em microdurômetro digital (FM 700, Future-Tech, Japão) com aumento de 500X para determinação do modo de fratura. As falhas poderão ser classificadas em: CD) falhas coesivas em dentina, quando ocorrerem exclusivamente em dentina; CC) falhas coesivas em compósito restaurador, quando ocorrerem em resina composta; A) falhas adesivas, quando ocorrerem na região de interface dentina-resina; M) falhas mistas, quando a falhas acontecem na interface adesiva, estendendo-se para um dos substratos vizinhos; FP) falhas prematuras, quando as falhas ocorrerem durante o preparo ou armazenamento dos espécimes.

5.8 Tratamento estatístico

O número de repetições em cada avaliação terá como base o cálculo amostral executado previamente a partir de um estudo piloto, adotando-se o poder de teste próximo ou superior a 80%. A partir dos resultados obtidos, o método estatístico será escolhido com base na aderência ao modelo de distribuição normal e igualdade de variância. Para todos os testes, será considerada significância estatística quando $\alpha=0.05$.

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ORÇAMENTO

Descrição	Quantidade	Custo (unidade)	Custo (total)
Acetona	1000 ml	R\$ 18,00	R\$ 18,00
Adesivo a base de cianoacrilato	10 un.	R\$ 5,00	R\$ 50,00
Adper Scotch Bond (3M ESPE)	1 kit	R\$ 160,00	R\$ 160,00
All Bond 2 (Bisco)	1 kit	R\$ 420,00	R\$ 420,00
Cera pegajosa em bastão	4 cx.	R\$ 25,00	R\$ 100,00
Compósito restaurador	10 un.	R\$ 50,00	R\$ 500,00
Disco de precisão para corte	1 un.	R\$ 1.600,00	R\$ 1.600,00
Étanol	1000 ml	R\$ 10,00	R\$ 10,00
HEMA	500 g	R\$ 350,00	R\$ 350,00
Impressões	10 un.	R\$ 50,00	R\$ 500,00
Lixas para desgaste (#600)	30 un.	R\$ 1,00	R\$ 30,00
Luvras de procedimento	1 cx.	R\$ 18,00	R\$ 18,00
Máscaras descartáveis	1 cx.	R\$ 10,00	R\$ 10,00
Opti Bond FL (Keer)	1 kit	R\$ 86,00	R\$ 86,00
Pincel descartável	1 cx.	R\$ 10,00	R\$ 10,00
Serviço de revisão do inglês	1 revisão	R\$ 200,00	R\$ 200,00
THF	1000 ml	R\$ 40,00	R\$ 40,00
		Total	R\$ 4.102,00

Quadro 1 - Orçamento previsto para viabilização do projeto.

CRONOGRAMA

Mês/Ano	Revisão de literatura	Teste piloto	Qualificação do projeto	Fase experimental	Análise estatística	Redação	Defesa de dissertação	Submissão do artigo para publicação
Fevereiro/08	X							
Março/08	X	X						
Abril/08	X		X					
Mai/08	X			X		X		
Junho/08	X					X		
Julho/08	X					X		
Agosto/08	X							
Setembro/08	X							
Outubro/08	X							
Novembro/08	X			X	X	X		
Dezembro/08	X					X		
Janeiro/09	X					X		
Fevereiro/09							X	
Março/09								X

Quadro 2 - Cronograma de execução do projeto.

ARTIGO²

² Artigo redigido segundo o Guia para Autores do Periódico Dental Materials disponível em: http://www.elsevier.com/wps/find/journaldescription.cws_home/601024/authorinstructions. Acesso em: 20 fev. 2009.

Tetrahydrofuran as alternative solvent in dental adhesive systems

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Abstract

Objective. To evaluate the influence of tetrahydrofuran (THF) on the resin-dentin microtensile bond strength (μ TBS) after water storage for 24 hours and 6 months and to compare it behavior with traditional solvents.

Methods. Seven versions of monomer/solvent mixtures were prepared using the following solvents and water combinations: 1) THF, 2) acetone, 3) ethanol, 4) water, 5) THF/water, 6) acetone/water and 7) ethanol/water. Forty-two bovine incisors, randomly separated into seven groups, had their superficial coronal dentin exposed. After acid-etching and rinse, the excess of water was removed from the surface with absorbent paper. Each solvent-based experimental primer was applied with agitation (30 s) followed by a gently air stream (10 s). The experimental adhesive resin was applied and light-activated (20 s). Resin composite restorations were constructed incrementally. Restored teeth were stored in distilled water at 37 °C (24 h) and sectioned to obtain sticks (0.5 mm²). Half of specimens were subjected to the μ TBS test immediately after cut and the other half was tested after water storage for 6 months. Data (MPa) were analyzed by two-way ANOVA, Tukey test and paired T-test at $\alpha = 0.05$.

Results. Both 'solvent type' and 'storage time' affected the resin-dentin μ TBS. Significant decreases in μ TBS were observed for all the groups tested after 6 months, except for THF-based primer that resulted in more stable bonds over this period.

Significance. THF appears to be a promising solvent to be used in dental adhesive systems, improving the resistance of resin-dentin bond to water degradation.

Keywords: Adhesive, bond strength, dentin, solvent, storage.

1. Introduction

Considerable number of bonding systems currently in use contain hydrophilic resin monomers often dissolved in volatile solvents such as acetone, ethanol, water or a combination of them [1]. The main objectives of the inclusion of these elements in etch-and-rinse adhesives formulations were to improve the dentin hybridization [2] and the net expansion of dried demineralized dentin matrix [3-7]. Because of the low molecular weight and high diffusion capacity, the solvents also decreases the viscosity and increases the hydrophilicity of solutions [1, 3, 8]. Besides, organic solvents aids in the displacement of residual water that should be completely eliminated from the surfaces prior to curing [9-12].

The knowledge of chemical and physical properties of adhesive components is indispensable to understand or even predict their behavior. Some of the main characteristics of solvents are its boiling temperature, vapor pressure, H-bonding capacity and polarity [1]. The two first properties ensure good evaporation of the solvents after application [13, 14], but volatility may also influence the shelf life of adhesives [15]. The high ability to hydrogen bonding is desirable because, by this way, solvents can remove water from the demineralized dentin matrix and preserves the collagen interfibrillar spaces at the same time [3-7]. Besides, their polarity determines the solubility characteristics of molecules [1]. Previous works indicated that methanol can reexpand the collapsed network because of it higher ability to form H-bond with collagen than the peptides have for themselves, yet it is very volatile [5, 7], even so its not used commercially.

Increased concentrations of hydrophilic compounds have to be used in the composition of primer and/or adhesive resin to enhance their ability to infiltrate within the demineralized dentin [2]. Unfortunately, these products are prone to water

sorption that adversely affects the stability of resin-dentin interfaces [16-19]. Thus, contemporary bonding procedures ideally should be done in the absence of water, employing hydrophobic resins that are expected to be more stable over the long term [2, 20, 21]. As residual solvent or insufficiently removed surface water are considered degradation promotion factors [18, 21], the role of solvent type in their elimination from the substrate it has been recognized [14, 22, 23]. Moreover, prolonged air-drying [11, 14, 22, 24] and prolonged application-times [13] were already proposed to optimize the elimination of organic solvents and water, but proposals of changes in the solvent type are quite few.

Tetrahydrofuran (THF) is a heterocyclic organic and polar aprotic solvent that dissolves a wide range of polar and nonpolar compounds [25]. These features may justify why THF could possible be used in dental adhesives that combine hydrophobic and hydrophilic components [1]. Moreover, the volatile character of THF imply that this solvent is easy to be removed after application in teeth superficies, like traditional solvents already evaluated [13]. In spite of supposed advantages above-described, little information was founded about THF up to now [26].

Whereas its clearly necessary optimizing the formulations to minimize the adverse effects of some ingredients commonly used in current dental adhesives, the aim of this study was to evaluate the influence of THF as an alternative solvent in the primer composition of an experimental adhesive system on the resin-dentin microtensile bond strength (μ TBS) after 24 h and 6 months of water storage and, also, to compare it behavior with traditional solvents. The null hypothesis tested was that there was no influence on the μ TBS of different solvent-based experimental primers, regardless of the time of water storage.

2. Materials and methods

2.1 Materials` preparation:

Seven versions of solvent-based experimental primers were used in this study. The blends were formulated through the intensive mixture of 50 wt% 2-hydroxyethyl methacrylate (HEMA) (Aldrich; St Louis, MO, USA), 10 wt% phosphate monomer [27] and 40 wt% solvent (Labsynth, Diadema, SP, Brazil), as described in Table 1. The concentrations of experimental primers were based on a pilot study, which indicated that a solvent concentration of 40 wt% was required for each monomer blend (unpublished data). These organic solvents were used as they were received from the manufacturers, without any additional treatment. An experimental adhesive resin (AD-50, CDC-Bio, Pelotas, RS, Brazil) was also prepared by addition of methacrylates, photoinitiators and stabilizers. All the weight measurements were carried out isothermally at 23 °C (± 1) using an analytical balance (AG 200, Gehaka, São Paulo, SP, Brazil). The freshly prepared blends were ultra-sonicated for 15 min, to ensure homogeneity, and used until 24 h after the preparation.

2.2 Specimens` preparation:

Forty-two extracted bovine incisors, that were disinfected in 0.5 % chloramine-T and used within 3 months after extraction, were chosen to this study. The teeth were randomly separated into seven experimental groups, assigning six teeth for each one. The superficial coronal dentin was exposed and further polished with a 600 grit silicon-carbide paper for 60 s to create a standardized smear layer.

All the bonding procedures were carried out by a single operator at a room temperature of 23 °C (± 1) and at a relative humidity of 70 % (± 5). After acid-etching with 35 % phosphoric acid for 15 s, the surfaces were rinsed with distilled water for 30 s and the excess of water was removed with absorbent paper. According the

group, one coat of the solvent-based experimental primer was applied to the dentin with agitation for 30 s using a brush. Air stream for 10 s at a distance of 10 cm was used to help solvent evaporation. One coat of an experimental adhesive resin was applied and light-activated for 20 s using a LED (Radii, SDI, Bayswater, Victoria, Australia) at 1.400 mW/cm^2 . Composite restorations were constructed on the bonded surfaces with four incremental layers of approximately 1.0 mm of resin composite (Charisma, Heraeus Kulzer, Hanau, Hesse, Germany). Each layer of composite was light-activated with the light unit set at the same time and intensity as above mentioned.

2.3 Microtensile bond strength test:

After storage in distilled water at 37°C for 24 h, the restored teeth were longitudinally sectioned in both “x” and “y” directions across the bonded interface with a diamond blade in a Isomet saw (Isomet 1000, Buehler, Lake Bluff, IL, EUA) at 400 rpm to obtain bonded sticks with a cross-sectional area of approximately 0.5 mm^2 . The cross-sectional area of each stick was measured with a digital caliper (DC 500, Mitutoyo, Suzano, SP, Brazil) and recorded for subsequent μTBS calculation. Approximately six sticks were produced from each tooth (eighteen sticks per group). Half of them were tested immediately after the cut and the other half were tested after 6 months of water storage at 37°C . Each stick was individually attached to a device for microtensile testing with cyanoacrylate resin (Loctite, Henkel, Diadema, SP, Brazil) and subjected to a tensile force in a universal testing machine (DL 500, Emic, São José dos Pinhais, PR, Brazil) at a cross-head speed of 1.0 mm/min . Bond strengths values, in mega-Pascal (MPa), were obtained by dividing the force, in Newton (N), by the calculated area, in mm^2 . Data records were submitted for statistical analysis (SigmaStat 3.1, Systat, Richmond, CA, USA), using two-way

ANOVA, Tukey test and paired T-test. The level of confidence was established at $\alpha = 0.05$.

2.4 Failure pattern analyzes:

The half corresponding to dentin in each specimen was removed from the device for microtensile testing and evaluated at 500 X in a light microscope (FM 700, Futuretech, Kawasaki, Tokyo, Japan). The failure patterns were classified as: cohesive in dentin (failure exclusive within dentin), cohesive in composite (failure exclusive within resin composite), adhesive (failure at resin-dentin interface), mixed (failure at resin-dentin interface that simultaneously included cohesive failure of the neighboring substrates) or premature debonded (failure during specimen preparation or water storage).

3. Results

The mean values and the standard deviations of resin-dentin microtensile bond strength are presented in Table 2 and Figure 1. Two-way analyzes of variance (solvent type versus storage time) showed significant differences for the factors 'solvent' and 'time' ($p < 0.001$). There was also a statistically significant interaction between these two factors ($p < 0.001$).

The mean values of μ TBS from the groups tested did not statistically differ after water storage for 24 h, except for the water-based primer that attained lower immediate μ TBS ($p < 0.001$). However, the only group which did not reduce bond strength was the THF-based primer, resulting in more stable bonds over 6 months ($p = 0.902$).

After water storage for 24 h, paired T-test presented no statistically significant difference between μ TBS of solvent-based primers and their corresponding with water ($p > 0.05$). Nevertheless, there were differences statistically significant between these same groups after 6 months evaluation, showing lower bond strengths in groups that contained water combination as solvent ($p < 0.05$).

Adhesive failures and premature debonded sticks were both associated with lower results of μ TBS. Sixteen out of the total number of tested specimens failed cohesively in resin composite. None specimen debonded prematurely while being prepared. On the other hand, there were eight premature debonded after storage for 6 months. The overall 6 months fracture analysis is expressed in Figure 2.

4. Discussion

The dependence of bond strength upon the composition of current etch-and-rinse adhesive systems to dentin was already demonstrated in recent studies [23, 28, 29]. However, when marketed available adhesives are compared, different components are appraised simultaneously. Moreover, manufacturers are usually reluctant to reveal the complete information of their products to avoid disclosure of the formulations [1]. The advantage of the present study was the use of experimental adhesive systems, which only differed on the type of solvent present on each primer. Thus, the authors expected to be able to determine the performance of THF-based primer in comparison to solvents traditionally available (i. e. acetone, ethanol, water or combinations).

According the methodology used in the present study, the surface dentin moisture and the primer application protocols were equally adopted for all experimental adhesive tested, regardless of the solvent type. In this manner, the solvent role in the degree of evaporation was assessed, trying to minimize variables. Although current adhesive systems require a moist dentin surface before bonding, it is know that the amount of water necessary to maximize bond strengths is different among the solvent-containing agent [4, 23, 29]. Despite the use of different artifices, such as prolonged application times and an air-dry stream, to improve the removal of residual water and organic solvents [13], it was reported that completely elimination is difficult to be achieved and dependent of the solvent type and content [14, 22, 23].

The present study required the rejection of the tested null hypothesis because demonstrated that both solvent type and storage time interfered in μ TBS results ($p < 0.001$). Despite statistically similar immediate μ TBS between THF (55.3 MPa), acetone (56.0 MPa) and ethanol (61.1 MPa) based primers, it was demonstrated that

these values were maintained only to THF-based primer (55.9 MPa) after 6 months of water storage. Additionally, the bond strength values suffered significant reduction for all the groups when water was added to the formulations after 6 months of evaluation ($p < 0.05$). This way, these data also confirms that most dental adhesive systems show favorable immediate bonding results, but the stability of the bonded interfaces still deserves major concerns [21].

Considering bond strength and further information of boiling temperature and vapor pressure [1, 15], the authors speculated that there might be a dependence between mechanical properties of the bonded interfaces and volatile character of solvents. Whereas, ethanol (43.9 mmHg) and water (17.5 mmHg) based primers present a lower vapor pressure and a higher difficulty to displace the remaining solvent and water [22]. Maybe these groups could achieve better μ TBS after 6 months if a drier dentin surfaces had been used [23, 29]. Additionally, solvents with high H-bonding capacity, like ethanol and water, preferentially interact with collagen peptides, preventing the collapse of the dentin matrix [3-5]. However, the water molecules can diffuse through the polymer matrices by binding successively to their polar sites via hydrogen bond [17].

Thus, storage time might be deleterious to the mechanical properties of some of the resin-dentin components, which could compromise their durability [16, 18, 19]. These findings could explain the worst performance after aging of groups that had water as solvent or combinations in their formulations. On the other hand, the high vapor pressure of THF (173 mmHg), very similar to acetone (184 mmHg), certainly must ensure enhanced evaporation of water left in dentin and, consequently, bond strength stability over the time. Besides that, these polar aprotic solvents do not have the required hydroxyl-group to form strong hydrogen bonds as mentioned before [1].

Adhesive failures and premature debonded sticks were both associated with lower μ TBS, especially when water was incorporated to formulations. Regarding to bond strength, they seem to rely on the quality of the hybrid layer [30], a poor-hybridized dentin has worst interfacial mechanical properties to resist the degradation. These findings cope with other researches which demonstrated that the retained organic solvent and water within the adhesive resin may severely compromise the bond strength of resin-dentine and affect the structural integrity of the hybrid layer [16, 19]. Besides, these lowest values of μ TBS indicate that the excess of water is more detrimental for water-based than to the other solvent-based adhesives [23], resulting in deterioration of the bonded interfaces even for short time period (i.e. 6 months).

Considering scarce information about the knowledge concerning THF in dental materials [26], further citotoxicity tests and long-term evaluations (up to one year) are necessary to verify its behavior as solvent in the composition of dental adhesive systems.

5. Conclusions

Within the scope of this investigation, it was concluded that THF is seen as an alternative solvent to be added in primer composition of dental adhesive systems to improve the bonding stability over the time. The null hypothesis was rejected because the present results showed that the type of solvent affected the bond strength to dentin and it also influenced the stability of the bond interface.

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Tables and figures

Table 1. Composition (wt%) of the experimental primers

Reagent Group	HEMA	Phosphate monomer	THF	Acetone	Ethanol	Water	Total
THF	50	10	40	0	0	0	100
Acetone	50	10	0	40	0	0	100
Ethanol	50	10	0	0	40	0	100
Water	50	10	0	0	0	40	100
THF/water	50	10	20	0	0	20	100
Acetone/water	50	10	0	20	0	20	100
Ethanol/water	50	10	0	0	20	20	100

HEMA: 2- hydroxylethyl methacrylate; THF: tetrahydrofuran.

Table 2. Mean values followed by standard deviations of bond strength values (in MPa) after 24 h and 6 months water storage for adhesive systems formulated by different solvent types

Groups	Storage period	
	24 hours	6 months
THF	^A 55.3 (13.2)a	^A 55.9 (9.7)a
Acetone	^A 56.0 (14.3)a	^B 46.5 (12.8)ab
Ethanol	^A 61.1 (11.4)a	^B 36.9 (19.3)bc
Water	^A 42.0 (12.1)b	^B 17.5(11.5)d

Different superscript capital letters indicate statistically significant differences among means in rows and different lower letters indicate statistically significant differences among means in columns ($p < 0.05$).

Figure 1. Means and standard deviations of bond strength values (in MPa) after 24 h and 6 months water storage for adhesive systems formulated by combination of different solvents and water

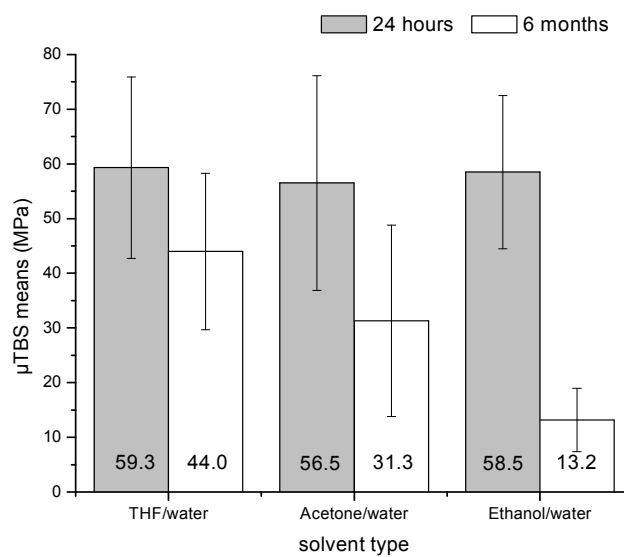
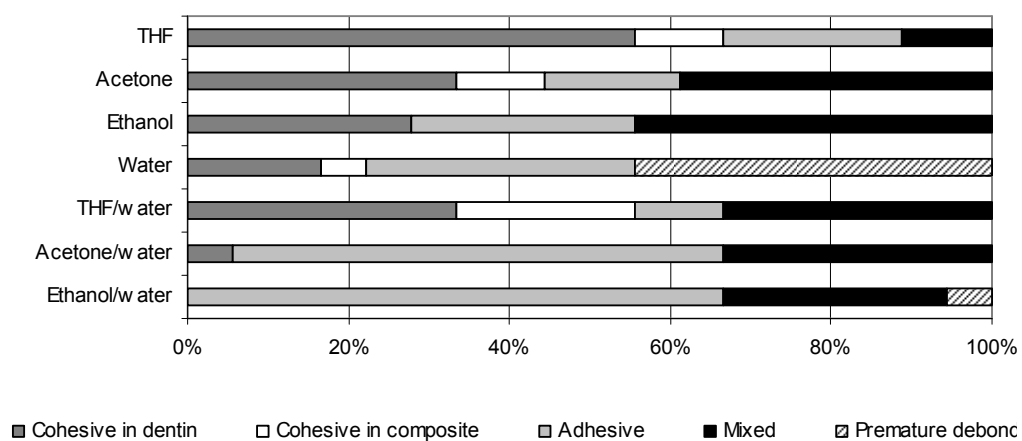


Figure 2. Failure pattern analyzes of 6 months evaluation



APÊNDICES

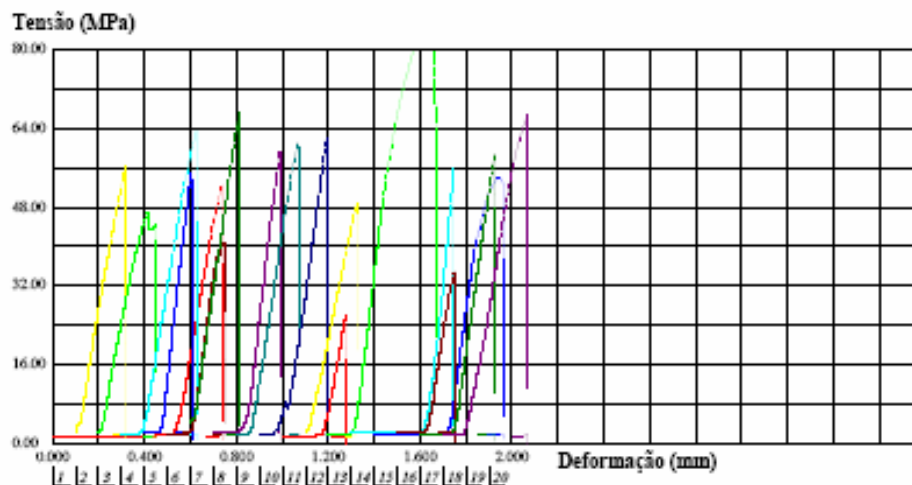
APÊNDICE A – Relatórios de ensaios mecânicos

UFPEL - Treinamento Evandro

Relatório de Ensaio

[illegible]

Corpo de Prova	Largura (mm)	Espessura (mm)	Área (mm ²)	Força Máxima (kgf)	Tensão Máxima (MPa)
CP 1	0.65	0.78	0.51	2.71	52.45
CP 2	0.65	0.66	0.43	2.47	56.37
CP 3	0.70	0.76	0.53	2.54	46.91
CP 4	0.66	0.67	0.44	2.87	63.58
CP 5	0.58	0.61	0.35	1.99	55.05
CP 6	0.61	0.66	0.40	1.67	40.74
CP 7	0.57	0.77	0.44	3.01	67.18
CP 8	0.59	0.62	0.37	2.22	59.47
CP 9	0.66	0.76	0.50	3.13	61.12
CP 10	0.62	0.74	0.46	2.90	61.96
CP 11	0.66	0.81	0.53	1.43	26.20
CP 12	0.60	0.81	0.49	2.43	48.94
CP 13	0.60	0.77	0.46	4.01	85.21
CP 14	0.54	0.65	0.35	2.00	55.88
CP 15	0.61	0.71	0.43	2.38	53.90
CP 16	0.58	0.64	0.37	1.32	34.97
CP 17	0.62	0.69	0.43	2.57	58.97
CP 18	0.69	0.71	0.49	3.35	67.07
Número CPs	18	18	18	18	18
Média	0.6217	0.7122	0.4437	2.500	55.33
Mediana	0.6150	0.7100	0.4406	2.505	56.12
Desv. Padrão	0.04328	0.06486	0.05851	0.6792	13.17
Coef. Var. (%)	6.963	9.106	13.19	27.17	23.79
Mínimo	0.5400	0.6100	0.3510	1.324	26.20
Máximo	0.7000	0.8100	0.5346	4.014	85.21



APÊNDICE B – Relatórios de análises estatísticas

Two Way Analysis of Variance

Data source: THF, ACE, ETH, WAT

General Linear Model

Dependent Variable: MPA

Normality Test: Passed (P = 0,887)

Equal Variance Test: Passed (P = 0,281)

Source of Variation	DF	SS	MS	F	P
SOLVENTE	3	11566,162	3855,387	21,488	<0,001
TEMPO	1	6949,817	6949,817	38,735	<0,001
SOLVENTE x TEMPO	3	3776,246	1258,749	7,016	<0,001
Residual	129	23144,881	179,418		
Total	136	42635,605	313,497		

The difference in the mean values among the different levels of SOLVENTE is greater than would be expected by chance after allowing for effects of differences in TEMPO. **There is a statistically significant difference (P = <0,001).** To isolate which group(s) differ from the others use a multiple comparison procedure.

The difference in the mean values among the different levels of TEMPO is greater than would be expected by chance after allowing for effects of differences in SOLVENTE. **There is a statistically significant difference (P = <0,001).** To isolate which group(s) differ from the others use a multiple comparison procedure.

The effect of different levels of SOLVENTE depends on what level of TEMPO is present. **There is a statistically significant interaction between SOLVENTE and TEMPO. (P = <0,001)**

Power of performed test with alpha = 0,0500: for SOLVENTE : 1,000

Power of performed test with alpha = 0,0500: for TEMPO : 1,000

Power of performed test with alpha = 0,0500: for SOLVENTE x TEMPO : 0,963

Least square means for SOLVENTE :

Group	Mean	SEM
THF	55,606	2,232
ACE	51,232	2,232
ETH	49,019	2,232
WAT	29,738	2,563

Least square means for TEMPO :

Group	Mean	SEM
24h	53,617	1,579
6m	39,181	1,700

Least square means for SOLVENTE x TEMPO :

Group	Mean	SEM
THF x 24h	55,332	3,157
THF x 6m	55,881	3,157
ACE x 24h	56,010	3,157

ACE x 6m	46,455	3,157
ETH x 24h	61,122	3,157
ETH x 6m	36,917	3,157
WAT x 24h	42,005	3,157
WAT x 6m	17,471	4,039

All Pairwise Multiple Comparison Procedures (Tukey Test):

Comparisons for factor: SOLVENTE

Comparison	Diff of Means	p	q	P	P<0,050
THF vs. WAT	25,868	4	10,763	<0,001	Yes
THF vs. ETH	6,587	4	2,951	0,158	No
THF vs. ACE	4,374	4	1,959	0,509	Do Not Test
ACE vs. WAT	21,495	4	8,943	<0,001	Yes
ACE vs. ETH	2,213	4	0,991	0,897	Do Not Test
ETA vs. WAT	19,281	4	8,022	<0,001	Yes

Comparisons for factor: TEMPO

Comparison	Diff of Means	p	q	P	P<0,050
24h vs. 6m	14,436	2	8,802	<0,001	Yes

Comparisons for factor: TEMPO within THF

Comparison	Diff of Means	p	q	P	P<0,05
6m vs. 24h	0,549	2	0,174	0,902	No

Comparisons for factor: TEMPO within ACE

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	9,555	2	3,026	0,032	Yes

Comparisons for factor: TEMPO within ETH

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	24,205	2	7,667	<0,001	Yes

Comparisons for factor: TEMPO within WAT

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	24,534	2	6,768	<0,001	Yes

Comparisons for factor: SOLVENTE within 24h

Comparison	Diff of Means	p	q	P	P<0,05
ETH vs. WAT	19,117	4	6,055	<0,001	Yes
ETH vs. THF	5,790	4	1,834	0,565	No
ETH vs. ACE	5,112	4	1,619	0,662	Do Not Test
ACE vs. WAT	14,005	4	4,436	0,009	Yes
ACE vs. THF	0,678	4	0,215	0,999	Do Not Test
THF vs. WAT	13,327	4	4,221	0,015	Yes

Comparisons for factor: SOLVENTE within 6m

Comparison	Diff of Means	p	q	P	P<0,05
THF vs. WAT	38,410	4	10,596	<0,001	Yes
THF vs. ETH	18,964	4	6,007	<0,001	Yes
THF vs. ACE	9,426	4	2,985	0,150	No

ACE vs. WAT	28,984	4	7,996	<0,001	Yes
ACE vs. ETH	9,538	4	3,021	0,142	No
ETH vs. WAT	19,446	4	5,365	<0,001	Yes

A result of "Do Not Test" occurs for a comparison when no significant difference is found between two means that enclose that comparison. For example, if you had four means sorted in order, and found no difference between means 4 vs. 2, then you would not test 4 vs. 3 and 3 vs. 2, but still test 4 vs. 1 and 3 vs. 1 (4 vs. 3 and 3 vs. 2 are enclosed by 4 vs. 2: 4 3 2 1). Note that not testing the enclosed means is a procedural rule, and a result of Do Not Test should be treated as if there is no significant difference between the means, even though one may appear to exist.

Two Way Analysis of Variance

Data source: THF/wat, ACE/wat, ETH/wat

General Linear Model

Dependent Variable: sqrt(col(3))

Normality Test: Passed (P = 0,3)

Equal Variance Test: Passed (P = 0,1)

Source of Variation	DF	SS	MS	F	P
SOLVENTE	2	40,3	20,2	15,5	<0,001
TEMPO	1	151,0	151,0	115,9	<0,001
SOLVENTE x TEMPO	2	40,1	20,0	15,4	<0,001
Residual	101	131,6	1,3		
Total	106	358,0	3,4		

The difference in the mean values among the different levels of SOLVENTE is greater than would be expected by chance after allowing for effects of differences in TEMPO. **There is a statistically significant difference (P = <0,001).** To isolate which group(s) differ from the others use a multiple comparison procedure.

The difference in the mean values among the different levels of TEMPO is greater than would be expected by chance after allowing for effects of differences in SOLVENTE. **There is a statistically significant difference (P = <0,001).** To isolate which group(s) differ from the others use a multiple comparison procedure.

The effect of different levels of SOLVENTE depends on what level of TEMPO is present. **There is a statistically significant interaction between SOLVENTE and TEMPO. (P = <0,001)**

Power of performed test with alpha = 0,05: for SOLVENTE : 1,0

Power of performed test with alpha = 0,05: for TEMPO : 1,0

Power of performed test with alpha = 0,05: for SOLVENTE x TEMPO : 1,0

Least square means for SOLVENTE :

Group	Mean	SEM
THF/wat	7,1	0,2
ACE/wat	6,4	0,2
ETH/wat	5,6	0,2

Least square means for TEMPO :

Group	Mean	SEM
24h	7,5	0,2
6m	5,2	0,2

Least square means for SOLVENTE x TEMPO :

Group	Mean	SEM
THF/wat x 24h	7,6	0,3
THF/wat x 6m	6,5	0,3
ACE/wat x 24h	7,4	0,3
ACE/wat x 6m	5,4	0,3
ETH/wat x 24h	7,6	0,3
ETH/wat x 6m	3,6	0,3

All Pairwise Multiple Comparison Procedures (Tukey Test):

Comparisons for factor: SOLVENTE

Comparison	Diff of Means	p	q	P	P<0,05
THF/wat vs. ETH/wat	1,5	3	7,9	<0,001	Yes
THF/wat vs. ACE/wat	0,7	3	3,6	0,034	Yes
ACE/wat vs. ETH/wat	0,8	3	4,3	0,008	Yes

Comparisons for factor: TEMPO

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	2,4	2	15,2	<0,001	Yes

Comparisons for factor: TEMPO within THF/wat

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	1,1	2	4,1	0,005	Yes

Comparisons for factor: TEMPO within ACE/wat

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	2,0	2	7,5	<0,001	Yes

Comparisons for factor: TEMPO within ETH/wat

Comparison	Diff of Means	p	q	P	P<0,05
24h vs. 6m	4,0	2	14,8	<0,001	Yes

Comparisons for factor: SOLVENTE within 24h

Comparison	Diff of Means	p	q	P	P<0,05
THF/wat vs. ACE/wat	0,2	3	0,8	0,828	No
THF/wat vs. ETH/wat	0,04	3	0,1	0,995	Do Not Test
ETH/wat vs. ACE/wat	0,2	3	0,7	0,875	Do Not Test

Comparisons for factor: SOLVENTE within 6m

Comparison	Diff of Means	p	q	P	P<0,05
THF/wat vs. ETH/wat	3,0	3	10,9	<0,001	Yes
THF/wat vs. ACE/wat	1,1	3	4,2	0,010	Yes
ACE/wat vs. ETH/wat	1,8	3	6,7	<0,001	Yes

A result of "Do Not Test" occurs for a comparison when no significant difference is found between two means that enclose that comparison. For example, if you had four means sorted in order, and found no difference between means 4 vs. 2, then you would not test 4 vs. 3 and 3 vs. 2, but still test 4 vs. 1 and 3 vs. 1 (4 vs. 3 and 3 vs. 2 are enclosed by 4 vs. 2: 4 3 2 1). Note that not testing the enclosed means is a procedural rule, and a result of Do Not Test should be treated as if there is no significant difference between the means, even though one may appear to exist.

t-test**Data source:** Data 1 in Notebook 1**Normality Test:** Passed (P = 0,786)**Equal Variance Test:** Passed (P = 0,241)

Group Name	N	Missing	Mean	Std Dev	SEM
THF (24h)	18	0	55,332	13,165	3,103
THF/wat (24h)	18	0	59,279	16,572	3,906

Difference -3,947

t = -0,791 with 34 degrees of freedom. (P = 0,434)

95 percent confidence interval for difference of means: -14,085 to 6,191

The difference in the mean values of the two groups is not great enough to reject the possibility that the difference is due to random sampling variability. **There is not a statistically significant difference between the input groups (P = 0,434).**

Power of performed test with alpha = 0,050: 0,050

The power of the performed test (0,050) is below the desired power of 0,800. Less than desired power indicates you are more likely to not detect a difference when one actually exists. Be cautious in over-interpreting the lack of difference found here.

t-test**Data source:** Data 1 in Notebook 1**Normality Test:** Passed (P = 0,706)**Equal Variance Test:** Passed (P = 0,420)

Group Name	N	Missing	Mean	Std Dev	SEM
THF (6m)	18	0	55,881	9,718	2,291
THF/wat (6m)	18	0	43,982	14,345	3,381

Difference 11,898

t = 2,913 with 34 degrees of freedom. (P = 0,006)

95 percent confidence interval for difference of means: 3,599 to 20,198

The difference in the mean values of the two groups is greater than would be expected by chance; **there is a statistically significant difference between the input groups (P = 0,006).**

Power of performed test with alpha = 0,050: 0,768

The power of the performed test (0,768) is below the desired power of 0,800. Less than desired power indicates you are more likely to not detect a difference when one actually exists. Be cautious in over-interpreting the lack of difference found here.

t-test**Data source:** Data 1 in Notebook 1**Normality Test:** Passed (P = 0,538)**Equal Variance Test:** Passed (P = 0,202)

Group Name	N	Missing	Mean	Std Dev	SEM
ACE (24h)	18	0	56,010	14,312	3,373
ACE/wat (24h)	18	0	56,477	19,642	4,630

Difference -0,467

t = -0,0815 with 34 degrees of freedom. (P = 0,936)

95 percent confidence interval for difference of means: -12,108 to 11,175

The difference in the mean values of the two groups is not great enough to reject the possibility that the difference is due to random sampling variability. **There is not a statistically significant difference between the input groups (P = 0,936).**

Power of performed test with alpha = 0,050: 0,050

The power of the performed test (0,050) is below the desired power of 0,800. Less than desired power indicates you are more likely to not detect a difference when one actually exists. Be cautious in over-interpreting the lack of difference found here.

t-test**Data source:** Data 1 in Notebook 1**Normality Test:** Passed (P = 0,668)**Equal Variance Test:** Passed (P = 0,279)

Group Name	N	Missing	Mean	Std Dev	SEM
ACE (6m)	18	0	46,455	12,781	3,013
ACE/wat (6m)	18	0	31,341	17,480	4,120

Difference 15,114

t = 2,961 with 34 degrees of freedom. (P = 0,006)

95 percent confidence interval for difference of means: 4,742 to 25,486

The difference in the mean values of the two groups is greater than would be expected by chance; **there is a statistically significant difference between the input groups (P = 0,006).**

Power of performed test with alpha = 0,050: 0,785

The power of the performed test (0,785) is below the desired power of 0,800. Less than desired power indicates you are more likely to not detect a difference when one actually exists. Be cautious in over-interpreting the lack of difference found here.

t-test**Data source:** Data 1 in Notebook 1**Normality Test:** Passed (P = 0,551)**Equal Variance Test:** Passed (P = 0,560)

Group Name	N	Missing	Mean	Std Dev	SEM
ETA (24h)	18	0	61,122	11,374	2,681
ETA/wat (24h)	18	0	58,507	13,968	3,292

Difference 2,614

t = 0,616 with 34 degrees of freedom. (P = 0,542)

95 percent confidence interval for difference of means: -6,014 to 11,243

The difference in the mean values of the two groups is not great enough to reject the possibility that the difference is due to random sampling variability. **There is not a statistically significant difference between the input groups (P = 0,542).**

Power of performed test with alpha = 0,050: 0,050

The power of the performed test (0,050) is below the desired power of 0,800. Less than desired power indicates you are more likely to not detect a difference when one actually exists. Be cautious in over-interpreting the lack of difference found here.

t-test**Data source:** Data 1 in Notebook 1**Normality Test:** Failed (P < 0,050)

Test execution ended by user request, Rank Sum Test begun

Mann-Whitney Rank Sum Test**Data source:** Data 1 in Notebook 1

Group	N	Missing	Median	25%	75%
ETA (6m)	18	0	31,090	21,580	55,060
ETA/wat (6m)	17	0	11,350	10,192	15,512

T = 172,000 n(small)= 17 n(big)= 18 (P = <0,001)

The difference in the median values between the two groups is greater than would be expected by chance; **there is a statistically significant difference (P = <0,001).**

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