Universidade de Lisboa

Faculdade de Medicina Dentária

Effect of Chlorhexidine Incorporation on the Properties of Acrylic Reline Resins

Catarina Matos Pereira de Sousa

Dissertação

Mestrado Integrado em Medicina Dentária

2014

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Dissertação orientada pela Professora Doutora Maria Cristina Bettencourt Neves e coorientada pela Professora Doutora Ana Francisca Bettencourt

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Agradecimentos

A concretização desta dissertação resulta da colaboração de várias pessoas, a quem gostaria de dirigir os meus sinceros agradecimentos.

À Professora Doutora Maria Cristina Bettencourt Neves, Professora Auxiliar da disciplina de Prostodontia Removível da Faculdade de Medicina Dentária da Universidade de Lisboa, quero expressar os meus sinceros agradecimentos pelo constante incentivo, orientação científica, disponibilidade e dedicação, que muito contribuíram para a elaboração deste trabalho. Agradeço também por me ter guiado durante todo este processo com apoio e amizade e por me ter transmitido a sua paixão pela investigação, que foi determinante para a execução desta tese.

À Professora Doutora Ana Francisca Bettencourt, Professora Auxiliar de Dispositivos Médicos e Física da Faculdade de Farmácia da Universidade de Lisboa, pelos ensinamentos e acompanhamento na realização de parte do trabalho experimental e pela disponibilidade, apoio e incentivo com que sempre me orientou.

Ao Professor Doutor Jaime Portugal, Professor Associado, regente da disciplina de Biomateriais da Faculdade de Medicina Dentária da Universidade de Lisboa, pela disponibilidade e interesse sempre demonstrados e pelos conhecimentos transmitidos na área de biomateriais e de estatística.

Aos técnicos de prótese Leonor Mega e Tomás Pinela agradeço toda a ajuda prestada no decorrer do estudo nas instalações de Prótese Dentária na Faculdade de Medicina Dentária da Universidade de Lisboa.

Gostaria ainda de agradecer aos meus amigos e colegas pelo convívio, motivação e incentivo durante o meu percurso académico. Em especial às minhas colegas e amigas Adriana Neves, Catarina Cesário, Catarina Abrantes, Ingride Ribeiras e Natacha Sousa, por toda a amizade e apoio, e por acreditarem em mim, mesmo nos momentos mais difíceis.

À minha colega e amiga Joana Costa, com quem partilhei este projeto, o meu sincero agradecimento pela sua amizade, colaboração e incentivo.

A todos os docentes, alunos e funcionários da Faculdade de Medicina Dentária da Universidade de Lisboa que, direta ou indiretamente, contribuíram para a minha formação pessoal e profissional e pelo convívio e amizade demonstrados ao longo destes anos.

Ao meu amigo e namorado Daniel Quaresma, por nunca duvidar das minhas capacidades e por me apoiar incondicionalmente. A sua presença constante e o carinho com que sempre me distinguiu foram determinantes para a conclusão desta etapa do meu percurso académico.

Por último, dedico este trabalho à minha família, em especial aos meus pais, a quem devo um eterno agradecimento pelo incondicional amor, apoio e incentivo que me transmitiram em todos os momentos da minha vida e por me terem ensinado a lutar pela realização de todos os meus objetivos.

Resumo

A estomatite protética é uma condição frequente em pessoas que usam próteses removíveis. Apesar de a sua etiologia ser multifatorial, a infeção por espécies de *Candida*, especialmente *Candida albicans*, é considerada o principal fator etiológico. Outros fatores locais como a presença de biofilme, trauma, xerostomia, uso contínuo da prótese e alteração do pH salivar, também estão associados a esta patologia. A aderência de *C. albicans* a células hospedeiras ou polímeros, tais como as resinas acrílicas usadas nas próteses, é o primeiro passo, essencial e necessário, no sucesso da colonização e desenvolvimento da infeção. O desenvolvimento de estomatite protética é influenciado, entre outros fatores, pelos materiais das bases das próteses. Estes materiais, como as resinas acrílicas, representam um suporte perfeito para a formação de biofilmes.

As abordagens terapêuticas existentes para o tratamento de lesões da mucosa oral são ineficazes, principalmente devido a dificuldades na aplicação da quantidade adequada do fármaco no local pretendido, bem como em manter o agente antimicrobiano na boca durante o tempo necessário para que o seu potencial terapêutico máximo seja atingido. Atualmente, a aplicação de fármacos é realizada através de um regime periódico não específico, por via tópica ou sistémica. Este método pode levar a efeitos secundários indesejáveis, devido a flutuações nos níveis farmacológicos.

A impregnação de dispositivos médicos com agentes antimicrobianos tem sido sugerida como tendo um potencial efeito na prevenção da aderência microbiana, o primeiro passo para a formação do biofilme. A libertação localizada e controlada destes agentes a partir do material também pode, potencialmente, inibir a maturação do biofilme. Estes sistemas de veiculação de fármacos permitem a libertação do agente no local da infeção com risco mínimo de níveis subterapêuticos ou toxicidade sistémica.

A clorexidina é um agente antimicrobiano vastamente prescrito como um colutório antisséptico em medicina dentária devido à sua atividade antimicrobiana de largo espetro, incluindo *C. albicans*. A sua incorporação em resinas acrílicas tem-se mostrado eficaz na supressão da capacidade da *C. albicans* para aderir às células epiteliais orais, e tem sido demonstrado que, quando incorporada em resinas acrílicas, a clorexidina é libertada a partir das mesmas, com uma taxa de eluição inicial elevada seguida de um processo de libertação controlada que continua durante os 28 dias de duração dos estudos.

A concentração de clorexidina que tem sido demonstrada como sendo eficaz frente à *C. albicans*, quando incorporada em resinas acrílicas, é de 10% da massa do pó da resina. Efetivamente, mostrou melhores resultados do que fármacos como o fluconazol. No entanto, pouco se sabe sobre as repercussões desta incorporação nas propriedades mecânicas e de superfície destes materiais.

O principal objetivo deste estudo foi avaliar o efeito da incorporação de clorexidina na microdureza, resistência à flexão e energia de superfície de duas resinas acrílicas de rebasamento direto, Kooliner e Ufi Gel Hard, e de uma resina acrílica de rebasamento indireto, Probase Cold.

Foram preparadas amostras de cada material a partir de moldes retangulares de aço inoxidável e, nas amostras experimentais, incorporou-se clorexidina numa proporção de 10% da massa do pó da resina acrílica. Para os testes de microdureza e resistência à flexão, foram preparadas trinta e duas amostras (64×10×3.3 mm) de cada material. Metade destas amostras foram incorporadas com clorexidina (grupo experimental) e a outra metade foi preparada sem qualquer tratamento (grupo controlo). As amostras de ambos os grupos foram aleatoriamente divididas em dois grupos, um que foi mantido a 37ºC±2ºC durante 48±2h e depois foi testado (não envelhecidos, n=8) e o outro foi testado após um procedimento de envelhecimento por termociclagem, correspondente a três meses de variações de temperatura no meio oral (envelhecidos, n=8).

A microdureza de todas as amostras foi obtida a partir do teste de microdureza Knoop. Após testar a microdureza, todas as amostras foram submetidas a um teste de resistência à flexão de três pontos.

Para estudar a energia de superfície, foram preparadas catorze amostras (25×16×1 mm) de cada resina, sendo que metade destas amostras foram incorporadas com clorexidina (grupo experimental, n=7) e a outra metade não sofreu qualquer tipo de tratamento (grupo controlo, n=7).

Através do método da placa de Wilhelmy, foram medidos, em cada amostra, os ângulos de contacto com água destilada e com 1,2-propanodiol e os valores obtidos foram usados para determinar o valor da energia de superfície total (γ), bem como das suas componentes dispersiva (γ_d) e polar (γ_p).

Foi feita a análise descritiva dos valores de microdureza, de resistência à flexão, dos ângulos de contacto com água e 1,2-propanodiol e da energia de superfície total e

seus componentes, tendo sido determinados os valores de média, mediana, desvio padrão e máximo e mínimo.

Sendo que os dados não apresentavam uma distribuição normal para as variáveis em estudo, os resultados foram submetidos a testes não-paramétricos pelo método de Mann-Whitney. Em todos os testes estatísticos, foi considerado um nível de significância igual a 5%.

Para a microdureza, a incorporação de clorexidina levou a valores inferiores na resina Kooliner, tanto para os grupos envelhecidos (p<0,001) como para os não envelhecidos (p<0,05). No Ufi Gel Hard, as amostras incorporadas com clorexidina e submetidas à termociclagem mostraram valores inferiores comparando com o grupo controlo (p<0,001) e as amostras não envelhecidas mostraram valores mais elevados que o grupo controlo (p<0,05). Para o Probase Cold, a incorporação de clorexidina não provocou diferenças na microdureza nas amostras não envelhecidas (p>0,05), mas levou a valores inferiores nas amostras envelhecidas (p<0,05) comparando com o grupo controlo.

Relativamente à resistência à flexão, as amostras de Kooliner com clorexidina mostraram valores inferiores (p<0,001), tanto nos grupos envelhecidos como nos não envelhecidos. No Ufi Gel Hard, não ocorreram diferenças na resistência à flexão entre grupos (p>0,05) nas amostras não envelhecidas, mas nas amostras que sofreram termociclagem, o grupo com clorexidina mostrou valores inferiores quando comparados com o grupo controlo (p<0,001). As amostras de Probase Cold com clorexidina obtiveram valores de resistência à flexão mais reduzidos, tanto nos grupos sem termociclagem (p<0,001) como nos grupos com termociclagem (p<0,05).

Relativamente à energia de superfície, a incorporação de clorexidina nas amostras de Kooliner levou a valores mais elevados de energia de superfície total e da componente polar (p=0,001). Não se verificaram diferenças significativas na componente dispersiva (p=0,805) entre grupos de Kooliner. Nas amostras de Ufi Gel Hard incorporadas com clorexidina, verificaram-se valores mais elevados de energia de superfície total (p=0,011) e da componente dispersiva (p=0,007), comparados com o grupo controlo, não se verificando diferenças significativas na componente polar (p=0,620) entre grupos. Nas amostras de Probase Cold, verificaram-se valores mais elevados de energia de superfície total (p=0,011) nos grupos com clorexidina, não se encontrando diferenças significativas nas componentes dispersiva (p=0,259) e polar (p=0,073).

Os resultados obtidos neste estudo mostram que a incorporação de clorexidina afeta a microdureza, a resistência à flexão e a energia de superfície das resinas acrílicas de rebasamento estudadas, sendo que o efeito varia com a resina avaliada.

De uma forma geral, o presente estudo dá a conhecer os efeitos da incorporação de clorexidina nas propriedades físicas dos materiais avaliados. São necessários mais testes, como por exemplo testes de resistência ao corte e de rugosidade de superfície, de forma a obter mais informação sobre quais as propriedades que sofrem alterações com a incorporação de clorexidina. Seria útil a realização de ensaios microbiológicos para se determinar se numa concentração inferior a clorexidina é eficaz contra a *C. albicans*. Sugere-se, ainda, a realização de testes de biocompatibilidade, de forma a avaliar se este fármaco, bem como a sua interação com a resina acrílica, podem ter efeitos tóxicos.

Palavras-chave: Incorporação de fármacos; Microdureza; Resistência à flexão; Energia de superfície; Resinas acrílicas.

Abstract

The main purpose of this study was to evaluate the effect of chlorhexidine incorporation on microhardness, flexural strength and surface free energy of three acrylic reline resins, Kooliner, Ufi Gel Hard and Probase Cold.

For all tests, half of the samples were incorporated with chlorhexidine 10% (*w/w*) and the other half were left unloaded. Both experimental and control specimens used for microhardness and flexural strength tests were randomly divided into two groups, one suffered a thermocycling aging process and the other didn't.

Specimens with 64×10×3.3 mm dimensions (n=8) were submitted to Knoop hardness and flexural strength tests; specimens with 25×16×1 mm dimensions (n=7) were submitted to contact angle measurements by Wilhelmy plate technique in order to obtain surface free energy values.

Data were submitted to nonparametric tests according to the Mann-Whitney method (p<0.05).

Chlorhexidine incorporation caused different effects, depending on the resins evaluated. On Kooliner, it led to lower microhardness and flexural strength values and to higher values for the total surface free energy, at cost of the increased values of the polar component. For Ufi Gel Hard, chlorhexidine incorporation caused higher microhardness values when no aging process was applied and on lower values on aged specimens. It also caused lower flexural strength values when thermocycling was applied. Regarding total surface free energy, higher values were found on chlorhexidine groups as a result of higher values of the dispersive component. Chlorhexidine incorporation on Probase Cold led to lower microhardness values on thermocycling groups and lower flexural strength values both when submitted to thermocycling and when not. It also resulted on slightly higher total surface free energy values, with no significant differences on the dispersive and polar components.

Overall, important insights of the chlorhexidine incorporation effects on the physical properties of the acrylic resins were known.

Keywords: Drug incorporation; Microhardness; Flexural strength; Surface energy; Acrylic resins.

1. Introduction

Tooth loss is a significant problem in the elderly population (Minami *et al.*, 2004) and at the present, an aging population increases the number of edentulous and partially dentate patients (Harwood, 2008). So, and according to a study performed by Douglass *et al.* (2002) in the United States, the number of people who need complete dentures will increase over the next 20 years despite an anticipated decline in the age-specific rates of edentulism (Douglass *et al.*, 2002). Therefore, the usage of removable dental prostheses, partial as well as complete, will be still a necessity for many people (Kranjcic *et al.*, 2013).

Denture stomatitis is a common condition among people who wear complete dentures (Ruby and Barbeau, 2002; Coco *et al.*, 2008). Although the etiology of denture stomatitis is multifactorial, infection by *Candida* species, especially *Candida albicans*, is considered the main etiologic factor (Ruby and Barbeau, 2002; Ramage *et al.*, 2006; Dagistan *et al.*, 2009; Redding *et al.*, 2009).

Local factors associated with the denture, such as presence of biofilm, local trauma, xerostomia, continuous use of the dentures and alteration in salivary pH, are also related to this pathology (Coco *et al.*, 2008; Chopde *et al.*, 2012).

Adherence of *C. albicans* to host cells or polymers, such as denture acrylic resins, is an essential and necessary first step in successful colonization and development of infection (Waters *et al.*, 1997). Development of denture stomatitis is influenced by the denture base material, among other factors (Coco *et al.*, 2008; Redding *et al.*, 2009; Chopde *et al.*, 2012).

Materials for prostheses, such as acrylic resins, represent a perfect support for biofilm formation. The chemical and physical characteristics of the surface of these materials support biofilm formation through reversible and then irreversible adhesion to the surface (Ramage *et al.*, 2006; Marra *et al.*, 2012).

The existing therapeutic approaches for the treatment of the oral mucosal lesions are highly inefficient. This is mainly due to difficulties in placing an adequate amount of the drug at the intended site as well as being then able to maintain the antimicrobial agent in the mouth sufficiently long for its maximum therapeutic potential to be achieved. Presently, the delivery of drugs is obtained by nonspecific periodic application of the agent to the organism, either topically or systemically. This method can lead to undesired side-effects, either at the target site or in the environment around

the target, due to fluctuation in drug levels (Darwish *et al.*, 2011; Salim *et al.*, 2012a; Salim *et al.*, 2013a; Sivakumar *et al.*, 2013).

Antimicrobial impregnation of medical devices has been suggested to have potential for the prevention of microbial adherence, the first step of biofilm formation. Slow release of the antimicrobial from the material also potentially inhibits biofilm maturation (Donlan, 2001). Incorporation of antibiotics into cements used in orthopedic surgery is a common practice (Frutos *et al.*, 2010; Matos *et al.*, 2014). The use of such drug delivery systems allows continuous drug release to the site of infection with minimal risk of subtherapeutic levels or systemic toxicity. Moreover, the use of self-releasing systems requires minimal intervention and monitoring (Salim *et al.*, 2012a).

Chlorhexidine (CHX) is an antimicrobial agent widely prescribed as an antiseptic mouthwash in dentistry due to its broad-spectrum antimicrobial activity, including *C. albicans* (Ryalat et al., 2011).

The antifungal effect of CHX incorporated in acrylic resins has been shown in many studies, and demonstrated that exposure of *C. albicans* to CHX suppresses its ability to adhere to buccal epithelial cells. Effectively, it has shown better results than other drugs like fluconazole, both on releasing and microbiological tests (Amin et al., 2009; Ryalat et al., 2011; Salim et al., 2013a; Salim et al., 2013b). Some studies have evaluated the CHX release from acrylic resins and concluded that there is a high initial rate of elution from material during the first 2–7 days, followed by a controlled sustained elution process that continues throughout the 28-day test period (Amin *et al.*, 2009; Ryalat *et al.*, 2011; Salim *et al.*, 2013a). Nevertheless, it wasn't found in the literature how much time it takes for CHX to fully disappear from the material.

The CHX concentration that has been shown to be the most effective against *C. albicans*, when incorporated in acrylic resins, is 10% (*w/w*) (Amin *et al.*, 2009; Ryalat *et al.*, 2011; Salim *et al.*, 2012a; Salim *et al.*, 2013a). However, scarce literature was found showing the repercussion of this CHX incorporation on the surface and mechanical properties of acrylic reline resins, both on immediate conditions and after CHX has been completely eluted from these materials.

The main purpose of this study was to evaluate the effect of CHX incorporation on microhardness, flexural strength and surface free energy of acrylic resins reline resins.

2. Objectives

The objective of this study was to evaluate the effect of CHX incorporation on the microhardness, flexural strength and surface free energy of acrylic reline resins, according to the following hypotheses:

H0: incorporation of CHX doesn't affect the microhardness of the reline resins.
H1: incorporation of CHX affects the microhardness of the reline resins.

H0: incorporation of CHX doesn't influence the flexural strength of the reline resins.
H1: incorporation of CHX influences the flexural strength of the reline resins.

H0: the surface free energy of the reline resins isn't affected by the incorporation of CHX.
H1: the surface free energy of the reline resins is affected by the incorporation of CHX.

3. Materials and Methods

Three auto-polymerizing acrylic resins (Table 3.1), presented in the powder-liquid form, were selected because of the differences in their chemical composition. Two of the acrylic resins are direct reline resins: a non-crosslinking material, Kooliner (GC America Inc, Alsip, Illinois, USA) (Figure 3.1a), and a crosslinking material, Ufi Gel Hard (Voco GmbH, Cuxhaven, Germany) (Figure 3.1b), composed of pre-polymerized poly(ethyl methacrylate) (PEMA) powder particles and the monomers isobutylmethacrylate (IBMA) or 1,6-hexanodioldimethacrylate (1.6-HDMA), respectively. One indirect reline resin, Probase Cold (Ivoclar Vivadent AG, Liechenstein) (Figure 3.1c) was used and represents a poly(methyl methacrylate) (PMMA) based material which has methylmethacrylate (MMA) as the monomer (Arima *et al.*, 1995 and 1996).

Table 3.1 – Materials under evaluation in the study.

Product	Manufacturer	Batch number	P/L ratio (g/mL)	Composition	Curing cycle
Kooliner (K)	GC America Inc., Alsip, Illinois, USA	1007201(P) 1008101(L)	1.4/1	P: PEMA L: IBMA	10 minutes 37ºC
Ufi Gel Hard (U)	Voco GmbH, Cuxhaven, Germany	1128441(P) 1134070(L)	1.77/1	P: PEMA L: HDMA	7 minutes 37ºC
Probase Cold (PC)	Ivoclar Vivadent AG, Liechtenstein	L49853(P) L43809(L)	1.5/1	P: PMMA L: MMA	15 minutes 40ºC 2-4 bar

P - Powder, L - Liquid, PEMA - polyethyl methacrylate, IBMA – isobutyl methacrylate, HDMA - hexanediol dimethacrylate PMMA - polymethyl methacrylate, MMA - methyl methacrylate.

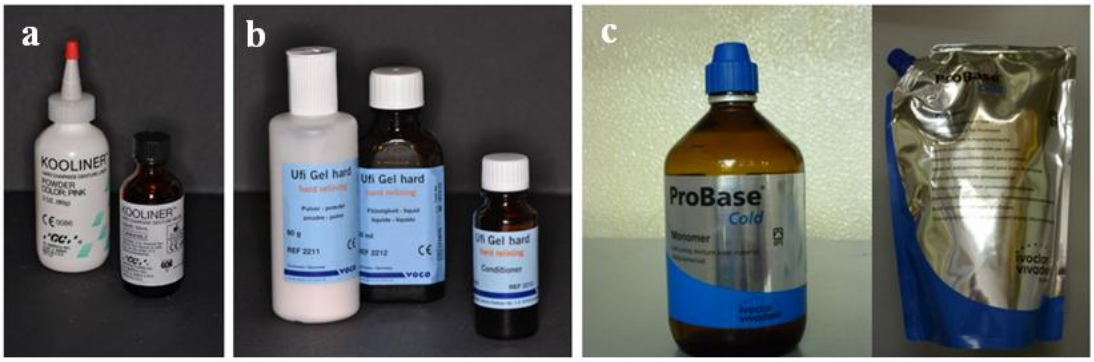

Figure 3.1 – Materials under evaluation in the study; a) Kooliner; b) Ufi Gel Hard; c) Probase Cold.

3.1 Preparation of the Specimens

The acrylic resins were manipulated according to the manufacturer's instructions (Table 3.1). The powder was weighed using a precision balance (Mettler Toledo) and the liquid was measured using a pipette. On the experimental specimens, chlorhexidine diacetate monohydrate (Panreac Applichem, Darmstadt, Germany) (CHX) (Figure 3.2a) at a proportion of 10% of the acrylic resin's powder weight (*w/w*) was incorporated and mixed using a mortar and pestle for homogenization (Figure 3.2b).

Specimens of each material were prepared from rectangular shaped stainless steel molds as ISO 20795-1 recommends (ISO 20795-1: 2013). The materials dough was maintained under compression at 37±2ºC, during the recommended polymerization time (Table 3.1) in order to simulate the intraoral polymerization of direct reline resins. Polymerization of the indirect reline resin was carried out in a pressure device (Ivomat, Ivoclar Vivadent, Liechenstein) (Figure 3.3) at recommended time, temperature and pressure (Table 3.1).

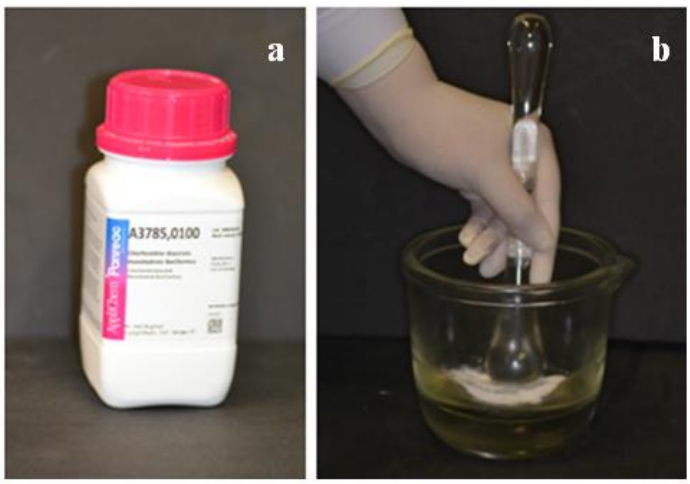

Figure 3.2 – Chlorhexidine diacetate monohydrate; a) Package; b) Incorporation and homogenization.

Figure 3.3 – Ivomat pressure device.

3.2 Microhardness and Flexural Strength Tests

Thirty-two samples (64×10×3.3 mm) of each material were prepared. Half of these samples were incorporated with CHX (experimental specimens) and the other half were left untreated (control group). On each preparation, the stainless steel mold was placed on a glass plate covered by a polyester sheet. The materials were prepared and placed into the mold. A new polyester sheet and glass plate were positioned on top of the mold and the set was maintained under compression (Figure 3.4). After polymerization, the samples were removed from the molds and the edges of each sample were polished with a 600-grit silicon carbide paper (Carbimet Paper Discs, Buehler Ltd., Lake Bluff, IL), on a polisher with constant refrigeration, in order to remove any irregularities (Figure 3.5).

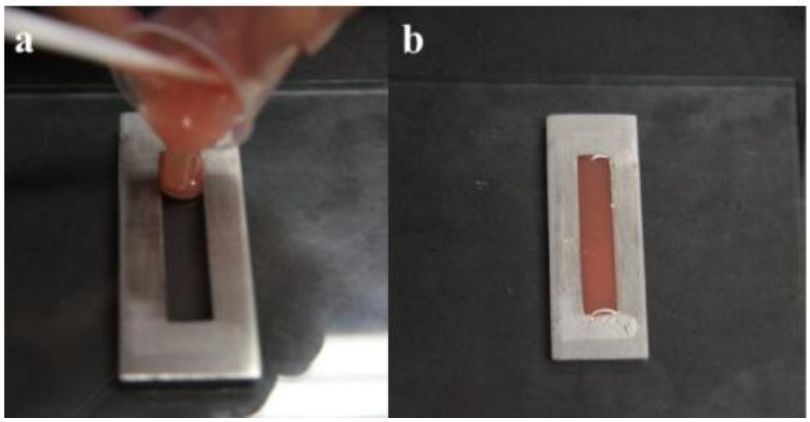

Figure 3.4 – Preparation of the specimens; a) Mixture of liquid and powder formulations is placed in the stainless steel mold; b) Mixture and mold between polyester sheets and glass plates.

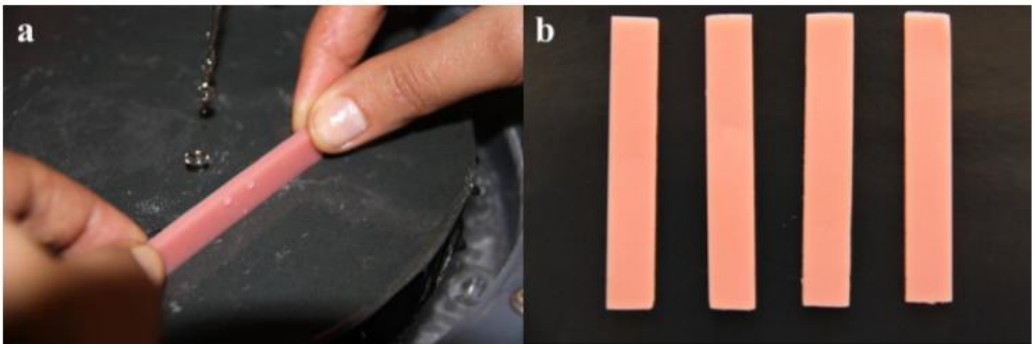

Figure 3.5 – Preparation of the specimens. After polymerization and removal of the specimen from the molds; a) Irregularities were removed; b) Examples of polymerized Kooliner specimens.

Both experimental and control specimens were randomly divided into two groups, one was kept at 37ºC±2ºC for 48±2h before testing (no aging, *n*=8) (ISO 20795-1: 2013) and the other one was tested after a thermocycling aging process (aging, *n*=8) as schematized on Table 3.2.

Table 3.2 – Schematization of distribution of the specimens.

Material	Conditions	CHX incorporation
Kooliner	No aging	Without CHX (*n*=8)
		With CHX (*n*=8)
	Aging	Without CHX (*n*=8)
		With CHX (*n*=8)
Ufi Gel Hard	No aging	Without CHX (*n*=8)
		With CHX (*n*=8)
	Aging	Without CHX (*n*=8)
		With CHX (*n*=8)
Probase Cold	No aging	Without CHX (*n*=8)
		With CHX (*n*=8)
	Aging	Without CHX (*n*=8)
		With CHX (*n*=8)

The aging samples were exposed to a thermocycling aging procedure of 2500 cycles of thermal fluctuations between 5ºC and 55ºC (20 seconds each bath), with 5 seconds of dwell time, in a specific machine (Refri 200-E, Aralab, Cascais, Portugal) (Figure 3.6).

Figure 3.6 – Thermocycling equipment.

3.2.1 Knoop Hardness Test

The microhardness of all samples was obtained using a Knoop diamond indentor, with an elongated pyramid's shape (Figure 3.7). The Knoop microhardness measurements were obtained by using a microhardness indention machine (Duramin, Struers DK 2750, Ballerup, Denmark), with a 98.12 mN load during 30 seconds (Pinto Lde *et al.*, 2010).

The operator, using the Duramin software, measured de length of the pyramids, immediately after each indentation, on a maximum period of ten seconds. Since there was a short time break between the indentation and the reading of the value, it was assumed that the viscoelastic recovery was minimal.

The equipment automatically converted these measurements into Knoop hardness numbers (KHN – kg/mm^2). Twelve measurements were made in each sample.

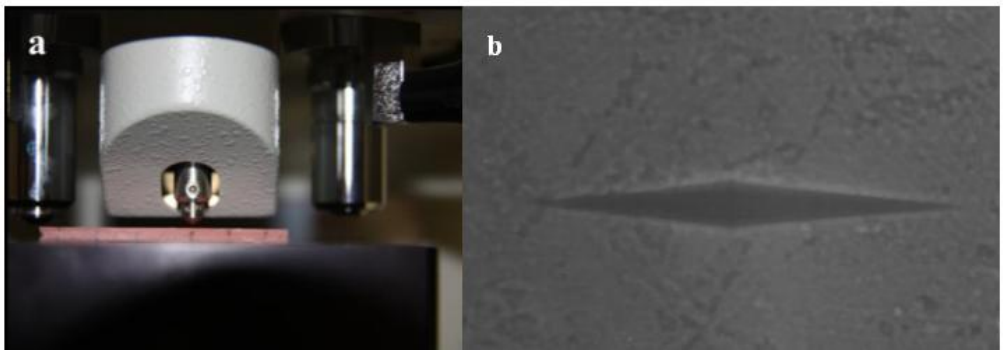

Figure 3.7 – a) Knoop indentor in a microhardness machine; b) microscopic image of a Knoop indentation on a Probase Cold specimen.

3.2.2 Flexural Strength Test

After microhardness testing, all specimens were submitted to the flexural strength test, in a servo-hydraulic universal machine (Instron Model 4502) (Figure 3.8) using three-point loading. A crosshead speed of 5mm per minute was used and the distance between supports was 50mm, as described elsewhere (ISO 20795-1: 2013). The specimen's dimensions (width and thickness) were measured using a digital micrometer (Mitutoyo Digimatic, MFG.Co., Ltd Tokyo, Japan) of 0.01mm precision and their averages were introduced in the software just before testing.

Load was applied until failure and the fracture load was recorded in Newtons (N). The flexural strength was expressed in megapascal (MPa) and calculated using the formula:

$$FS = \frac{3Wl}{2bd^2}$$

Where *FS* is the flexural strength, *W* is the maximum load before fracture (N), *L* is the distance between supports (50mm), *b* is the specimen's width (mm) and *d* is the specimen's thickness (mm).

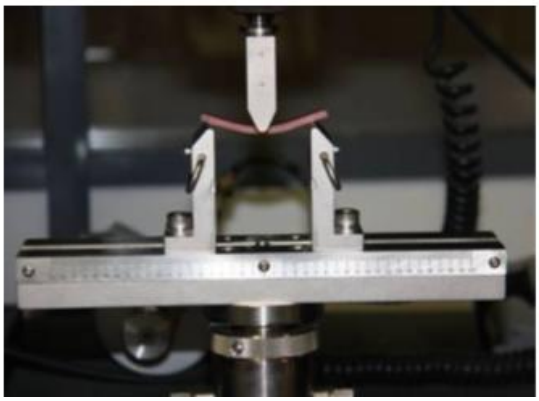

Figure 3.8 – Specimen submitted to 3 point loading flexural strength test in a universal machine.

3.3 Surface Free Energy

Specimens were obtained by packing the material's dough into rectangular metal molds (160×18×1 mm) and then each mold was clamped together in order to displace any material's excess (Figure 3.9a). After polymerization (Figure 3.9b), the samples were removed from the molds and were cut into different plates of approximate dimensions of 25mm width, 16mm height and 1mm thickness. The edges of each sample were polished with a 600-grit silicon carbide paper (Carbimet Paper Discs, Buehler Ltd., Lake Bluff, IL) in order to remove any irregularities. The specimens were

incubated at 37ºC±2ºC during 48±2h, immersed in distilled water, before the testing began.

Fourteen samples of each material were prepared. Half of these samples were incorporated with CHX (experimental group, *n*=7) and the other half was left untreated (control group, *n*=7).

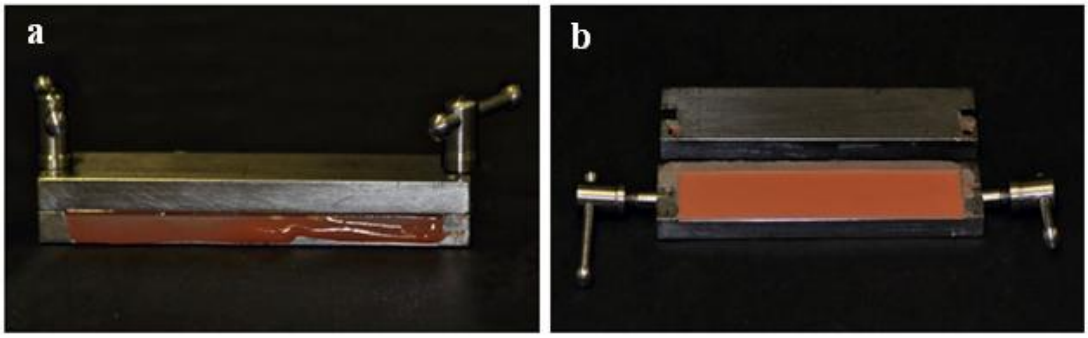

Figure 3.9 – a) Compression of resins dough in the metal mold; b) Metal mold opened after polymerization of the acrylic reline resin.

3.3.1 Contact Angle Test

To determine the surface free energy of the acrylic reline resin specimens, contact angles of distilled water and 1,2-propanediol were measured, using the Wilhelmy plate technique (Bettencourt *et al.*, 2004).

Testing was carried out using a Processor Tensiometer K12 (Kruss, Hamburg, Germany) linked to a computer (Figure 3.10).

Firstly, the specimen's dimensions (height, width and thickness) were measured using a digital micrometer (Mitutoyo Digimatic, MFG.Co., Ltd Tokyo, Japan) and introduced in the software just before testing. At the beginning of each experiment, a specimen of acrylic reline resin was suspended in the balance (sensitivity equal to 10^{-4} g) of the equipment. The system was set in a "Perspex®" box to ensure an artificially controlled environment. A glass cuvette containing the liquid under study (water or 1,2-propanediol) was placed in a steel container with thermostatic circulating water (25± 1ºC). Before changing the liquid, the cuvette glass was carefully washed with water and acetone mixture and was further assed into the flame of a Bunsen burner to reduce the likelihood of surface contamination.

A motorized platform allowed the immersion of 4mm of the specimen in the liquid, at a speed of 20 µm s^{-1} in the liquid (Figure 3.11).

In all the procedure, care was taken handling the specimens to reduce the chance of contamination of their surfaces.

Figure 3.10 – Processor Tensiometer K12: Equipment used in Wilhelmy plate technique.

3.3.2 Surface Free Energy Determination

Advancing contact angles were used for surface free energy (γ) estimation of all specimen, as well as its dispersive (γ_d) and polar components (γ_p) based on the harmonic mean method proposed by Wu (1971) (Wu, 1971). Equations for surface free energy estimation were solved using the equation handling KRUSS-software program: contact angle measuring system K121 (version 2.049) (Appendix 2, Figures 1 and 2).

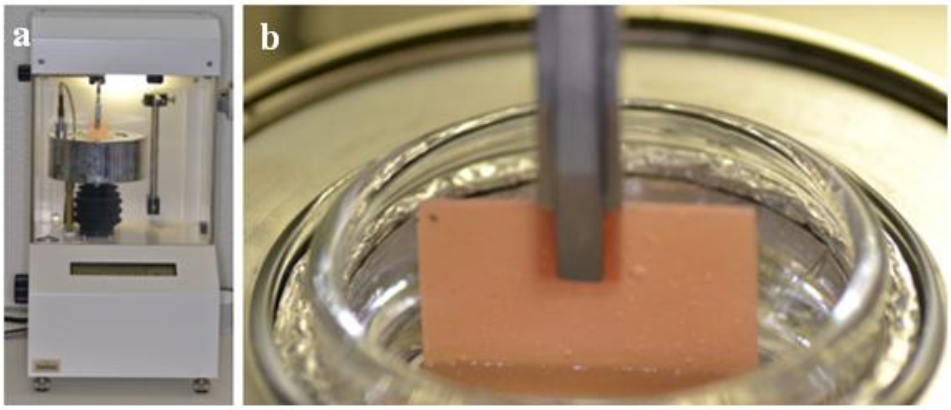

Figure 3.11 – a) Specimen of acrylic reline resin suspended in the balance of the equipment; b) Specimen of acrylic reline resin immersed in the glass cuvette with distilled water.

3.4 Statistical analysis

Descriptive statistics of microhardness, flexural strength, contact angle and surface free energy data was carried out. Mean, median, standard deviation and maximum and minimum values were determined.

Since data did not follow a normal distribution for the studied variables (verified by Kolmogorov-Smirnov normality tests), the results were submitted to the nonparametric tests according to the Mann-Whitney method.

In all statistical tests, it was considered the 5% level of significance ($p<0.05$).

Data were statistically analyzed using SPSS Statistics 20 (SPSS Inc., Chicago, IL, USA).

4. Results

For each material, the descriptive analysis of the data was carried out, including mean, median, standard deviation and maximum and minimum values for microhardness (Appendix 1, Table 1), flexural strength (Appendix 1, Table 2), contact angle (Appendix 1, Table 3) and surface free energy (Appendix 1, Table 4).

4.1 Effect of CHX incorporation on microhardness

Mean and standard deviation are graphically presented and explained by material (Figures 4.1-4.3).

For Kooliner specimens (Figure 4.1), CHX groups showed lower values of microhardness than the control groups, both in no aging ($p<0.05$) and aging groups ($p<0.001$).

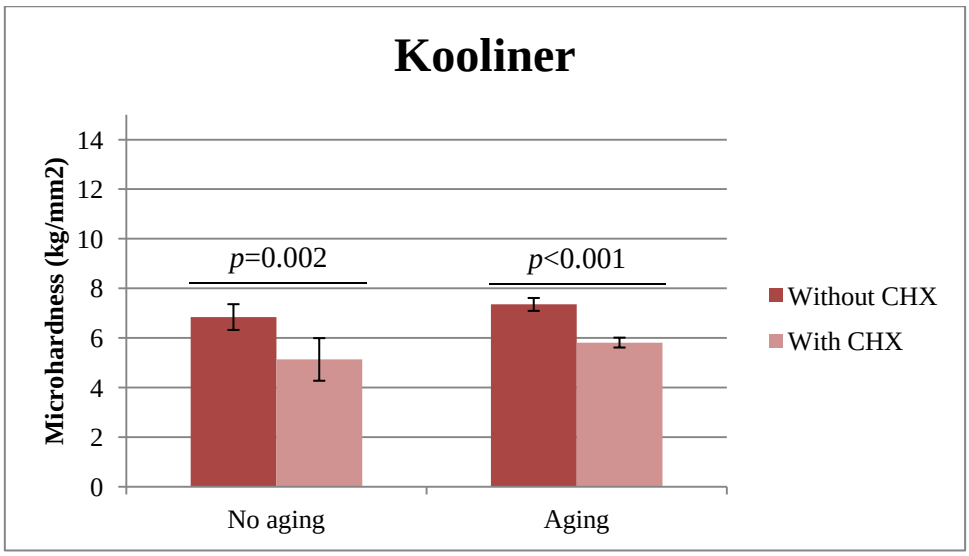

Figure 4.1 – Mean and standard deviation of values of microhardness (kg/mm^2) of Kooliner.

Ufi Gel Hard specimens (Figure 4.2) incorporated with CHX and submitted to aging showed lower values than the control group ($p<0.001$). The CHX group that was tested without thermocycling showed higher values than the control group ($p<0.05$).

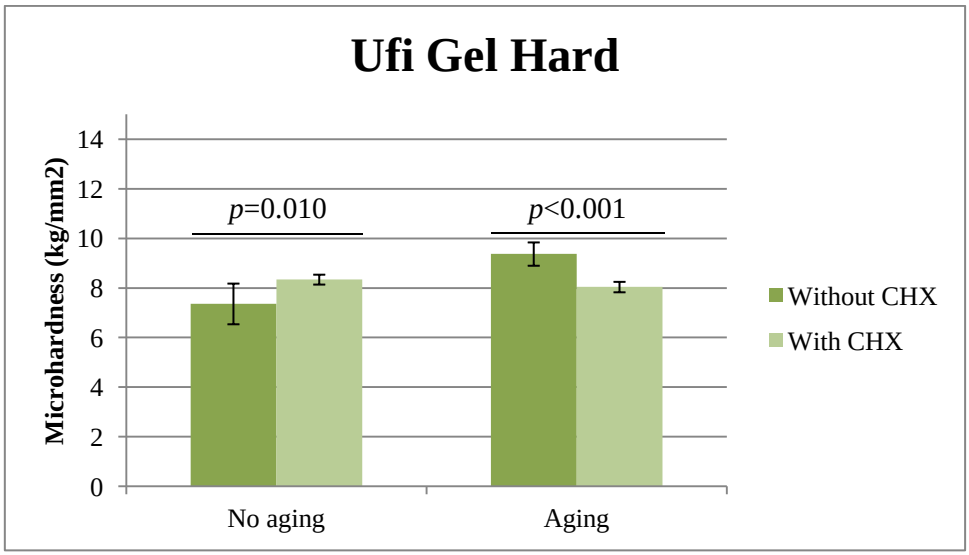

Figure 4.2 – Mean and standard deviation of values of microhardness (kg/mm^2) of Ufi Gel Hard.

On Probase Cold specimens (Figure 4.3), CHX incorporation led to no differences in microhardness in specimens not submitted to thermocycling ($p>0.05$). When specimens were submitted to aging, the CHX group showed lower values than the control group ($p<0.05$).

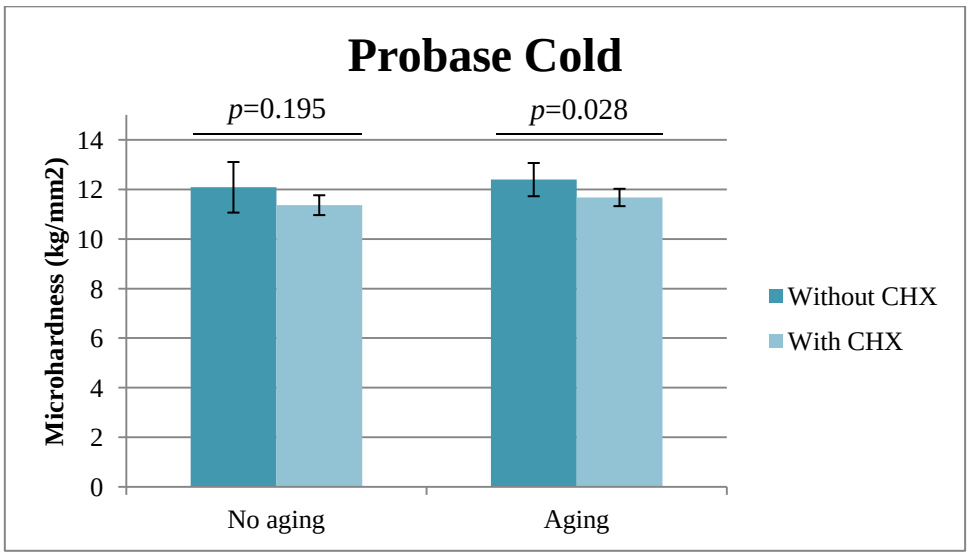

Figure 4.3 – Mean and standard deviation of values of microhardness (kg/mm^2) of Probase Cold.

4.2 Effect of CHX incorporation on flexural strength

Mean and standard deviation are graphically presented and explained by material (Figures 4.4-4.6).

Considering Kooliner specimens (Figure 4.4), CHX incorporation led to significant differences in flexural strength ($p<0.001$), both in no aging and aging conditions, with the CHX groups showing lower values than the control groups.

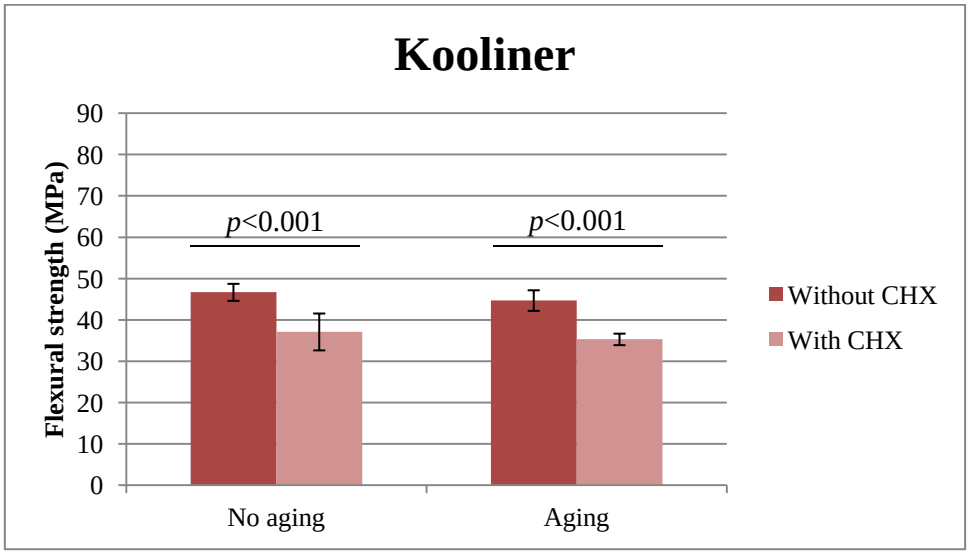

Figure 4.4 – Mean and standard deviation of values of flexural strength (MPa) of Kooliner.

On Ufi Gel Hard (Figure 4.5), there were no significant differences in flexural strength between groups ($p>0.05$) in no aging conditions but there were significant differences between groups when materials were submitted to a thermocycling treatment ($p<0.001$), with the CHX group showing lower values than the control group.

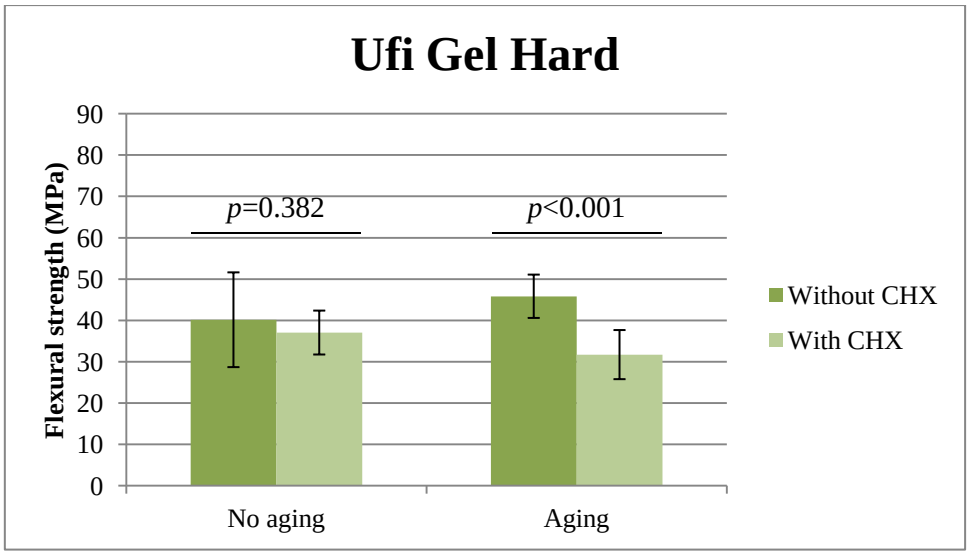

Figure 4.5 – Mean and standard deviation of values of flexural strength (MPa) of Ufi Gel Hard.

Probase Cold specimens (Figure 4.6) incorporated with CHX showed lower values than the control specimens, both when no aging process was carried ($p<0.001$) and when specimens were submitted to thermocycling ($p<0.05$).

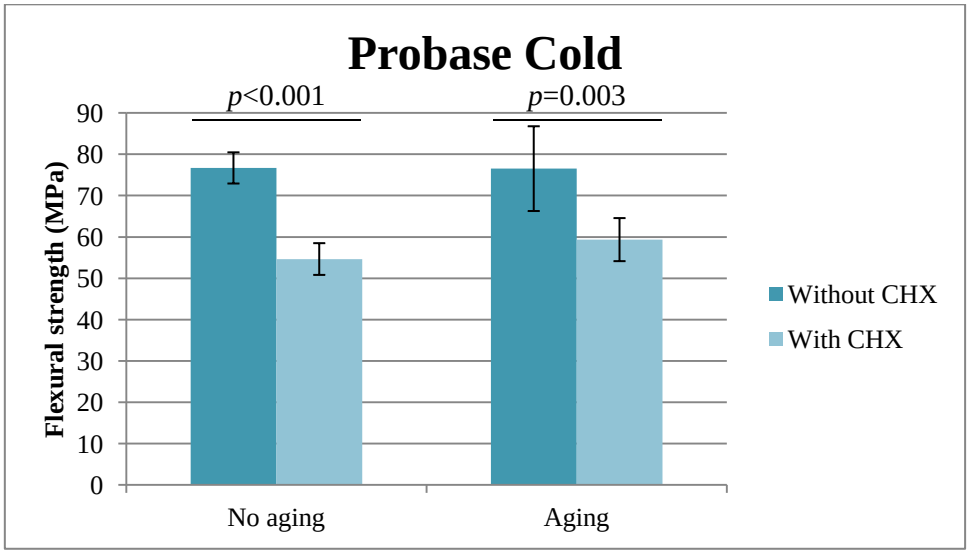

Figure 4.6 – Mean and standard deviation of values of flexural strength (MPa) of Probase Cold.

4.3 Effect of CHX incorporation on surface free energy

In order to study the effect of CHX incorporation on the surface free energy of acrylic reline resins, the experimental contact angle (water and 1,2-propanediol) values were used to estimate the total surface free energy and its dispersive and polar components. Mean and standard deviation of contact angle and surface free energy are presented on Table 4.1 and explained by material.

Table 4.1 – Mean and standard deviation values for contact angle and surface free energy of all materials.

Material	CHX incorporation	Contact angle (º)		Surface free energy (γ) (mN/m)		
		Water	1,2-propanediol	γ Total	γ Dispersive	γ Polar
Kooliner	Without CHX	94.45±1.84^{A}	53.41±2.99^{A}	25.80±0.71^{A}	17.06±1.92^{A}	8.76±1.47^{A}
	With CHX	74.29±1.86^{B}	37.28±5.70^{B}	35.86±1.52^{B}	17.40±1.59^{A}	18.46±1.01^{B}
Ufi Gel Hard	Without CHX	72.76±2.14^{A}	36.43±3.27^{A}	36.73±1.29^{A}	17.40±0.91^{A}	19.30±1.31^{A}
	With CHX	70.66±2.63^{A}	28.11±2.82^{B}	38.89±1.05^{B}	19.34±1.10^{B}	19.51±1.97^{A}
Probase Cold	Without CHX	75.52±4.42^{A}	39.68±4.17^{A}	35.08±2.09^{A}	17.02±1.51^{A}	18.07±2.95^{A}
	With CHX	69.08±4.42^{B}	32.43±3.57^{B}	39.09±2.58^{B}	17.91±0.72^{A}	21.17±2.64^{A}

Vertically identical superscripted capital letters denote no significant differences among groups (p>0.05)

Considering Kooliner specimens, significant differences in total surface free energy and in the polar component were found between groups (p=0.001), with the CHX group showing higher values than the control group. There were no significant differences in the dispersive component (p=0.805) between groups.

On Ufi Gel Hard specimens, significant differences in total surface free energy (p=0.011) and in dispersive component (p=0.007) were found, with the CHX groups presenting higher values than the control groups. There were no significant differences in the polar component (p=0.620).

For Probase Cold specimens, significant differences in total surface free energy were found (p=0.011), with the CHX group showing slightly higher values than the control group. No significant differences were found in the dispersive (p=0.259) and polar (p=0.073) components between groups.

5. Discussion

The addition of antimicrobial compounds to acrylic resins in order to create a slow drug releasing device has been widely evidenced in microbiological and release studies (Riggs *et al.*, 2000; Hiraishi *et al.*, 2008; Amin *et al.*, 2009; Redding *et al.*, 2009; Bettencourt *et al.*, 2010; Li *et al.*, 2010; Darwish *et al.*, 2011; Acosta-Torres *et al.*, 2012; Cochis *et al.*, 2012; Marra *et al.*, 2012; Salim *et al.*, 2012a; Salim *et al.*, 2013a; Salim *et al.*, 2013b).

However, there weren't found many studies regarding the evaluation of physical properties of these acrylic resins with antimicrobial agents incorporated. Some studies have investigated the peel bond strength (Alcantara *et al.*, 2012; Salim *et al.*, 2012b), hardness (Addy and Handley, 1981; Regis *et al.*, 2011), flexural strength (Casemiro *et al.*, 2008; Cunha *et al.*, 2009; Regis *et al.*, 2011; Sodagar *et al.*, 2013) and roughness (Cunha *et al.*, 2009; Regis *et al.*, 2011) of these acrylic resins, and from these, only Addy and Handley (1981), Alcantara *et al.* (2012) and Salim *et al*. (2012) studied the effect of CHX incorporation on acrylic resins.

The antifungal effect of CHX incorporated in acrylic resins has been investigated in many studies, revealing a more effective candidacidal effect compared to other drugs, such as fluconazole, both on releasing and microbiological tests (Amin et al., 2009; Ryalat et al., 2011; Salim et al., 2013a; Salim et al., 2013b). Some studies have evaluated the CHX release from acrylic resins and concluded that there is a high initial rate of elution from the material followed by a controlled sustained elution process that continues throughout the 28-day test period (Amin *et al.*, 2009; Ryalat *et al.*, 2011; Salim *et al.*, 2013a).

No studies were found in the literature that evaluated the effect of this incorporation on the surface and mechanical properties of acrylic reline resins after CHX has been completely eluted from these materials. Therefore, an aging thermocycling process corresponding to 3 months of temperature variation in the oral environment (Gale and Darvell, 1999) that can be induced by routine eating, drinking and breathing (Palmer *et al.*, 1992), was randomly applied on half of the specimens studied for microhardness and flexural strength. Since there weren't found studies that concluded when CHX had completely disappeared from the acrylic resin, the 3 months

period was chosen has an approximation of the time after which CHX is thought to have been completely eluted from the material.

In the present study, microhardness, flexural strength and surface free energy were evaluated.

Furthermore, differences between resins seemed interesting to study and statistical analysis between resins was carried out. Significant differences in microhardness values were found between all resins. Probase Cold showed significant higher values of flexural strength than the direct reline resins. Surface free energy values were also different between resins, with Kooliner revealing lower values than the other two materials. This is in agreement with a study by Arima (1995) that concluded that there are mechanical differences between cross-linking and non-crosslinking PEMA-based resins and between these and materials that contain mainly PMMA (Arima *et al.*, 1995). Also, the fact that the curing cycle of Probase Cold is carried out at higher temperature and pressure leads to a higher monomer conversion and therefore its mechanical properties should be improved (Urban *et al.*, 2007).

An important property, which makes it possible to use acrylic materials in dentures, is their hardness (Ali *et al.*, 2008; Pinto Lde *et al.*, 2010).

Hardness is defined as the resistance of a material to permanent surface indentation or penetration. A material with higher surface hardness could withstand excessive wear by denture cleanser, toothbrush, and food better than a softer material (Ali *et al.*, 2008).

CHX incorporation had different effects depending on the acrylic resin tested. On Kooliner, CHX incorporation led to lower microhardness values. Ufi Gel Hard specimens where CHX was incorporated showed higher microhardness values when no aging process was applied and lower values on specimens that suffered a thermocycling treatment. On Probase Cold, no differences were found between groups in non-aged specimens and lower microhardness values were showed on CHX groups submitted to thermocycling process.

All resins' results are in agreement with Addy and Handley (1981) that concluded that CHX incorporation reduced significantly the hardness values of the acrylic resins after 87 days of soaking in water (Addy and Handley, 1981). On the

present study, on aging conditions, the CHX incorporation groups showed significantly lower values than the control groups.

Studies that evaluated CHX release from acrylic resins have showed that this drug is liberated from the resin at least during 28 days after incorporation (Hiraishi *et al.*, 2008; Amin *et al.*, 2009; Salim *et al.*, 2012a). This may be one of the reasons that causes significantly lower values when CHX is incorporated in aging conditions.

If an acrylic resin presents lower hardness values, it means that it will probably be less resistant to some external agents like toothbrushes and food (Ali *et al.*, 2008). In this case, only Ufi Gel Hard and Probase Cold with CHX in no aging conditions showed significantly higher values and no differences, respectively. This means that these two materials could be used as carriers for local delivery of this drug within the oral cavity, if employed as reline materials in existing prostheses, with the probable necessity of being substituted after some time (Addy and Handley, 1981). It has been concluded before that Probase Cold has the lower levels of cytotoxicity, from the three tested acrylic resins (Mendes De Oliveira *et al.*, 2014), and this is a factor that may influence the decision when choosing between these two materials.

The flexural strength test has been constantly used in order to predict the material's behavior when submitted to masticatory forces (Haselton *et al.*, 2002; Balkenhol *et al.*, 2007; Ali *et al.*, 2008).

On flexural strength, CHX incorporation also had different effects depending on the acrylic resin tested. In Kooliner and Probase Cold, CHX groups showed lower flexural strength values both when submitted to thermocycling and when not submitted to this treatment. Ufi Gel Hard showed no significant differences between groups in no aging conditions, but CHX led to lower values when thermocycling was applied.

Kooliner and Probase Cold results are in agreement with other studies that evaluated the influence of antimicrobial agents' incorporation (silver-zinc zeolite, fluoroalkyl methacrylate, methacryloyloxyundecylpyridinium bromide or TiO_2 and SiO_2 nanoparticles) on acrylic resin's flexural strength, which have shown that the flexural strength was significantly lower after the incorporation of the compounds (Casemiro *et al.*, 2008; Cunha *et al.*, 2009; Regis *et al.*, 2011; Sodagar *et al.*, 2013).

Once again, the fact that CHX is continuously released from the acrylic resins, may cause a significantly decrease on flexural strength values when CHX is incorporated (Hiraishi *et al.*, 2008; Amin *et al.*, 2009; Salim *et al.*, 2012a).

If an acrylic resin presents lower flexural strength values, it means that it will be less resistant to some external agents like masticatory forces and have higher probability of suffering fractures (Haselton *et al.*, 2002; Balkenhol *et al.*, 2007; Ali *et al.*, 2008; Casemiro *et al.*, 2008). In the present study, all materials showed significantly lower values, except for Ufi Gel Hard in no aging conditions that showed no differences. This means that this material with CHX incorporated could be used as reline material but its risk of fracture will be increased after 3 months of use.

The total surface free energy of a solid is the sum of components arising from dispersive (apolar) and polar contributions. The contact angles formed with two liquids (water and 1,2-propanediol) on the acrylic resins' surface were used to calculate the surface free energy by the Wu method (Bettencourt *et al.*, 2004). The method enabled the calculation of the unknown solid surface energy components (polar and dispersive) from contact angle measurements with the two mentioned liquids (Waters and Jagger, 1999; Sipahi *et al.*, 2001; Zissis *et al.*, 2001; da Silva *et al.*, 2008).

Again, in the present study, CHX incorporation led to different effects depending on the acrylic resin tested. Kooliner was the acrylic resin that suffered more changes with this incorporation, with CHX groups showing higher values for the total surface free energy and the polar component. The dispersive component suffered no significant changes. With this information we can deduce that Kooliner surface becomes more polar when CHX is incorporated. For Ufi Gel Hard specimens, significant differences in total surface free energy and in dispersive component were found between groups, with the CHX groups showing higher values than the control groups. There were no significant differences in the polar component between groups. This means that this resin has a tendency to become more apolar with CHX incorporation. On Probase Cold, significant differences in total surface free energy were found between groups, with the CHX group showing significant higher values than the control group. There were no significant differences in the dispersive component and in the polar component between groups. This means that although the total surface free energy is different when CHX is incorporated, the balance between polar and dispersive components stays the same.

Changes in the surface free energy of the materials will directly impact its surface wettability. In sum, wettability can influence different aspects that are relevant

to materials performance, such as retention and stability of removable dentures, as well as adherence of microorganisms.

The retention and stability of removable dentures are related to various factors, such as wettability of denture base and denture relining materials, because it provides a condition in which saliva will easily spread over the surfaces (Zissis *et al.*, 2001; Combe *et al.*, 2004; Nishioka *et al.*, 2006; Jin *et al.*, 2009). In this case, CHX incorporation in Kooliner promoted higher values of total surface energy which means improved wettability. Therefore, this material with CHX incorporated would provide more retention and stability of removable dentures, improving patient's comfort.

As stated before, CHX is released from acrylic resins at least for 28 days after incorporation (Hiraishi *et al.*, 2008; Amin *et al.*, 2009; Salim *et al.*, 2012a) and this factor, allied to a higher polarity of Kooliner, may lead to an increasing water sorption (directly proportional to CHX releasing) and subsequently to worst physical properties.

The effect of wettability on the adherence of microorganisms as *Candida* is not consensual. Studies on denture-base materials have shown that there is a relationship between the cell numbers of *Candida* species adhering per unit area and the contact angle measurement. Some of these studies postulate that the more hydrophobic the surface the less the cell adherence (Minagi *et al.*, 1985; Al-Dwairi *et al.*, 2012). Alternatively, it has been proposed that there is higher adherence of *Candida* species in hydrophobic surfaces (Minagi *et al.*, 1985; Yoshijima *et al.*, 2010; Lazarin *et al.*, 2013). Furthermore, there are other studies that conclude that there is no conclusive relation between the material's surface free energy and the Candida adherence, and that the factor that contributes more to the adherence process is the cell's surface free energy, which can be different between species (Waters *et al.*, 1997; Webb *et al.*, 1998). In addition, other factors should also be considered, such as other cell surface factors, diet, salivary composition and secretion rates, and antibody titers, which are all controlling factors in plaque formation and could therefore influence yeast attachment (Al-Dwairi *et al.*, 2012).

The effect of the acrylic resin's surface free energy on *C. albicans* adhesion to these materials remains to be investigated. In order to determine if the surface modifications caused by CHX incorporation increase the *Candida* adherence other parameters must be evaluated, such as the surface roughness and microbiological assays.

6. Conclusions

With the results obtained in the present study, it can be concluded that CHX incorporation leads to different effects in microhardness, flexural strength and surface free energy, depending on the acrylic reline resins evaluated.

a. On Kooliner, CHX incorporation led to lower microhardness and flexural strength values in all tested conditions. It also increased the surface free energy of this resin due to higher values of its polar component.

b. For Ufi Gel Hard, although incorporation of CHX resulted on slightly higher microhardness values when no aging process was applied, it resulted on decreasing values of microhardness and flexural strength after aging. Regarding total surface free energy, higher values were found on CHX groups at cost of increasing values of the dispersive component.

c. CHX incorporation on Probase Cold led to lower flexural strength values as an immediate effect and after aging, with the latest being also characterized by decreasing microhardness values. In addition, it resulted on higher total surface free energy values, with no significant differences on the dispersive and polar components.

Overall, the present study can provide important insights into the CHX incorporation effects on the physical properties of the materials evaluated. Further studies will be necessary, such as shear bond strength and surface roughness tests, in order to obtain more information on which properties are affected by CHX incorporation. Microbiological assays would be useful to evaluate the effect of CHX incorporation on materials compliance *in vivo* and also to determine if a lower CHX concentration would be effective against *Candida* species. Biocompatibility studies as *in vitro* cytotoxicity assays are suggested, so that it is possible to conclude if this drug and its interaction with the acrylic resin could be harmful for human cells.

7. References

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Appendices

Appendix 1 – Tables

Table 1 – Mean, median, standard deviation, minimum and maximum values for microhardness (kg/mm^2).

Material	Conditions	CHX incorporation	*n*	Mean	Median	Standard deviation	Minimum	Maximum
Kooliner	No aging	Without CHX	8	6.84	6.66	0.52	6.37	7.85
		With CHX	8	5.13	4.95	0.86	4.02	6.66
	Aging	Without CHX	8	7.35	7.28	0.26	6.99	7.83
		With CHX	8	5.81	5.82	0.20	5.57	6.10
Ufi Gel Hard	No aging	Without CHX	8	7.36	7.33	0.82	6.14	8.98
		With CHX	8	8.34	8.35	0.20	7.93	8.61
	Aging	Without CHX	8	9.37	9.38	0.47	8.68	10.01
		With CHX	8	8.04	8.04	0.21	7.73	8.43
Probase Cold	No aging	Without CHX	8	12.08	11.90	1.02	10.88	13.69
		With CHX	8	11.36	11.33	0.40	10.92	12.00
	Aging	Without CHX	8	12.39	12.41	0.67	11.08	13.21
		With CHX	8	11.67	11.68	0.35	11.14	12.19

Table 2 – Mean, median, standard deviation, minimum and maximum values for flexural strength (MPa).

Material	Conditions	CHX incorporation	*n*	Mean	Median	Standard deviation	Minimum	Maximum
Kooliner	No aging	Without CHX	8	46.69	47.10	2.06	43.75	49.15
		With CHX	8	37.11	35.38	4.46	32.62	43.32
	Aging	Without CHX	8	44.70	45.18	2.49	40.54	47.58
		With CHX	8	35.31	35.77	1.39	32.68	37.06
Ufi Gel Hard	No aging	Without CHX	8	40.15	43.51	11.46	20.35	52.82
		With CHX	8	37.05	38.51	5.31	28.57	43.97
	Aging	Without CHX	8	45.83	45.44	5.24	39.75	56.87
		With CHX	8	31.72	31.78	5.94	22.33	40.87
Probase Cold	No aging	Without CHX	8	78.68	78.38	3.77	73.23	85.99
		With CHX	8	54.64	54.30	3.84	49.44	60.47
	Aging	Without CHX	8	76.51	76.81	10.25	61.12	92.25
		With CHX	8	59.34	59.31	5.21	50.60	67.20

Table 3 – Mean, median, standard deviation, minimum and maximum values for contact angle (º) with water and 1,2-propanediol.

Material	Liquid	CHX incorporation	*n*	Mean	Median	Standard deviation	Minimum	Maximum
Kooliner	Water	Without CHX	7	94.45	94.75	1.84	91.21	97.14
		With CHX	7	74.29	74.75	1.86	72.13	77.42
	1,2-propanediol	Without CHX	7	53.41	53.59	2.99	48.43	56.95
		With CHX	7	37.28	36.85	5.70	30.89	44.88
Ufi Gel Hard	Water	Without CHX	7	72.76	73.25	2.14	69.80	75.20
		With CHX	7	70.66	70.72	2.63	66.04	74.81
	1,2-propanediol	Without CHX	7	36.43	35.68	3.27	32.49	40.19
		With CHX	7	28.11	27.55	2.82	24.61	31.74
Probase Cold	Water	Without CHX	7	75.52	75.01	4.42	69.39	83.28
		With CHX	7	69.08	68.93	4.42	60.85	75.37
	1,2-propanediol	Without CHX	7	39.68	41.07	4.17	30.84	42.63
		With CHX	7	32.49	34.58	3.57	28.24	36.29

Table 4 – Mean, median, standard deviation, minimum and maximum values for surface free energy (total, dispersive and polar) (mN/m).

Material	γ	CHX incorporation	*n*	Mean	Median	Standard deviation	Minimum	Maximum
Kooliner	Total	Without CHX	7	25.80	25.70	0.71	24.70	27.10
		With CHX	7	35.86	36.40	1.52	33.30	37.60
	Dispersive	Without CHX	7	17.06	17.00	1.92	14.30	19.70
		With CHX	7	17.40	17.70	1.59	15.20	19.40
	Polar	Without CHX	7	8.76	8.60	1.47	6.80	11.30
		With CHX	7	18.46	18.60	1.01	17.00	20.00
Ufi Gel Hard	Total	Without CHX	7	36.73	36.70	1.29	35.00	38.30
		With CHX	7	38.89	38.90	1.05	37.60	40.70
	Dispersive	Without CHX	7	17.40	17.10	0.91	16.60	18.80
		With CHX	7	19.34	19.40	1.10	17.60	21.10
	Polar	Without CHX	7	19.30	18.80	1.31	17.90	21.10
		With CHX	7	19.51	19.60	1.97	16.40	23.10
Probase Cold	Total	Without CHX	7	35.08	35.50	2.09	31.40	37.90
		With CHX	7	39.09	39.40	2.58	35.70	43.90
	Dispersive	Without CHX	7	17.02	16.70	1.51	15.50	19.52
		With CHX	7	17.91	17.80	0.72	17.10	18.80
	Polar	Without CHX	7	18.07	17.70	2.95	13.50	22.30
		With CHX	7	21.17	20.80	2.64	17.30	26.10

Appendix 2 – Figures

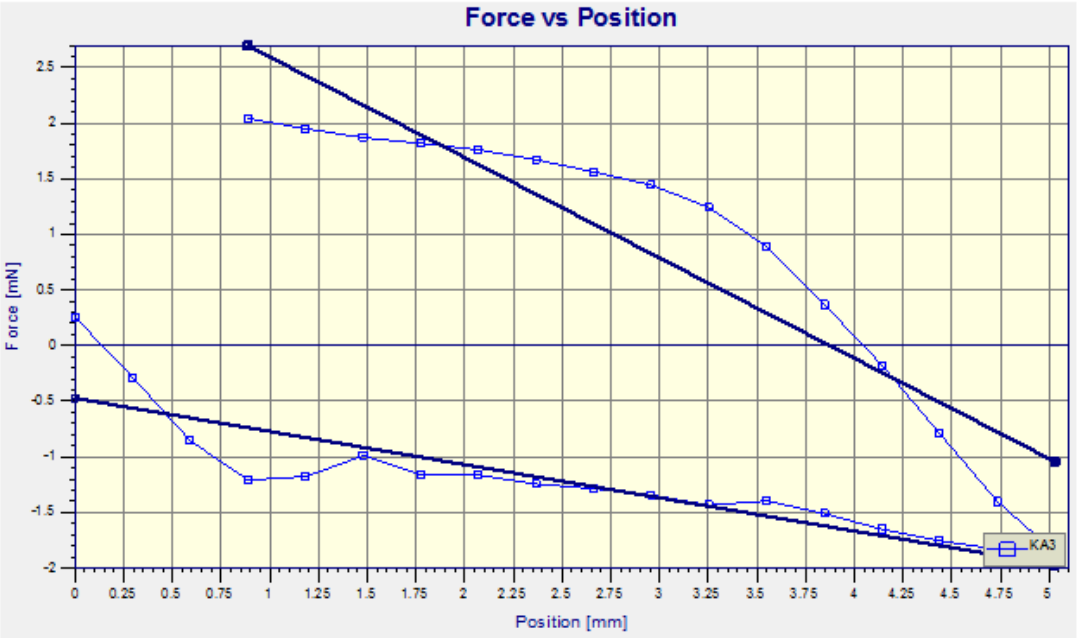

Figure 1 – One example of graphical obtained for determination of the contact angle of a Kooliner specimen.

RESULTS

Total number of liquids suitable for calculation: 2
Maximum number of liquid combinations : 1
Number of liquids combined and calculated : 1
Number of invalid liquids combinations : 0

LIQUID COMBINATION 1

MEASUREMENT	LIQUID	SFTDISPER: [mN/m]	[mN/
KA3PG	PG	38.0	28
KA3	Water	72.1	19

The calculation of the solid's free surface energy deliver:
(Harmonic-Mean Method):

	Solution 1	Solution 2
Surface energy of solid:	26.0 mN/m	-149.8 mN/m
disperse part:	19.3 mN/m	-143.4 mN/m
polar part:	6.8 mN/m	-6.4 mN/m

Figure 2 – One example of determination of the surface free energy of a Probase Cold specimen.

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Appendix 5 – List of Abbreviations

γ	Surface free energy
γ_d	Dispersive component of surface free energy
γ_p	Polar component of surface free energy
1,6-HDMA	1,6-hexanedioldimetacrylate
CA	Contact angle
CHX	Chlorhexidine diacetate monohydrate
FS	Flexural strength
HDMA	Hexanediol dimethacrylate.
IBMA	Isobutylmethacrylate
ISO	International Organization for Standardization
K	Kooliner
KH	Knoop hardness
KHN	Knoop Hardness Number
MMA	Methylmethacrylate
MPa	Megapascal
PC	Probase Cold
PEMA	Polyethilmethacrylate
PMMA	Polymethylmethacrylate
U	Ufi Gel Hard

Appendix 6 – Experimental Data

Knoop Hardness (Kooliner)

Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)
KA1.1	6,4	KB1.1	4,8	KAE1.1	8,2	KBE1.1	6,1
KA1.2	5,8	KB1.2	5,3	KAE1.2	8,1	KBE1.2	5,3
KA1.3	5,2	KB1.3	5,3	KAE1.3	7,9	KBE1.3	6
KA1.4	5,2	KB1.4	5,8	KAE1.4	7,5	KBE1.4	5,9
KA1.5	5,4	KB1.5	5,2	KAE1.5	6	KBE1.5	5,5
KA1.6	7,1	KB1.6	5,9	KAE1.6	6	KBE1.6	6,4
KA1.7	7,1	KB1.7	6,1	KAE1.7	5,6	KBE1.7	5,4
KA1.8	6,8	KB1.8	5	KAE1.8	7,3	KBE1.8	6,1
KA1.9	6,4	KB1.9	4,8	KAE1.9	6,9	KBE1.9	5,8
KA1.10	7,4	KB1.10	5,1	KAE1.10	6,6	KBE1.10	5,4
KA1.11	6,7	KB1.11	5,8	KAE1.11	9,2	KBE1.11	6,7
KA1.12	6,9	KB1.12	4,9	KAE1.12	10	KBE1.12	6,6
KA2.1	7,5	KB2.1	7,5	KAE2.1	8,1	KBE2.1	5,6
KA2.2	7,6	KB2.2	8,5	KAE2.2	6,9	KBE2.2	5,7
KA2.3	7,1	KB2.3	5,1	KAE2.3	10,4	KBE2.3	6,6
KA2.4	8,4	KB2.4	7	KAE2.4	9,2	KBE2.4	6,9
KA2.5	7,9	KB2.5	7	KAE2.5	6	KBE2.5	6,9
KA2.6	7,9	KB2.6	6,7	KAE2.6	6,8	KBE2.6	6,2
KA2.7	8,8	KB2.7	5,6	KAE2.7	6,5	KBE2.7	5,8
KA2.8	10,7	KB2.8	4,5	KAE2.8	7,5	KBE2.8	5,3
KA2.9	7,4	KB2.9	5,4	KAE2.9	7,8	KBE2.9	7,4
KA2.10	7,6	KB2.10	5,6	KAE2.10	8,3	KBE2.10	6
KA2.11	7	KB2.11	8,1	KAE2.11	6,7	KBE2.11	5,5
KA2.12	6,3	KB2.12	8,9	KAE2.12	6,7	KBE2.12	5,3
KA3.1	7,2	KB3.1	4,1	KAE3.1	9	KBE3.1	5,3
KA3.2	6,6	KB3.2	4	KAE3.2	7,2	KBE3.2	6,2
KA3.3	7,3	KB3.3	4	KAE3.3	7,1	KBE3.3	5,5
KA3.4	5,7	KB3.4	4	KAE3.4	6,4	KBE3.4	6,3
KA3.5	5,5	KB3.5	4,1	KAE3.5	6,5	KBE3.5	5,7
KA3.6	5,3	KB3.6	4	KAE3.6	7,2	KBE3.6	5,3
KA3.7	5,1	KB3.7	3,8	KAE3.7	7,9	KBE3.7	5,3
KA3.8	6,6	KB3.8	3,6	KAE3.8	7,6	KBE3.8	5,4
KA3.9	5,5	KB3.9	3,8	KAE3.9	6,9	KBE3.9	5,5
KA3.10	6,2	KB3.10	3,4	KAE3.10	6,4	KBE3.10	6,2
KA3.11	7,4	KB3.11	4,4	KAE3.11	6,4	KBE3.11	6,4
KA3.12	8,1	KB3.12	5	KAE3.12	8	KBE3.12	5,4
KA4.1	6,7	KB4.1	4,2	KAE4.1	8,1	KBE4.1	5,7
KA4.2	4,8	KB4.2	6,4	KAE4.2	7,7	KBE4.2	5,4
KA4.3	7,1	KB4.3	5,2	KAE4.3	8,3	KBE4.3	5,6
KA4.4	7,2	KB4.4	5,2	KAE4.4	8,4	KBE4.4	6,1
KA4.5	5,3	KB4.5	5,8	KAE4.5	8,3	KBE4.5	5,8
KA4.6	5,8	KB4.6	6,2	KAE4.6	7,3	KBE4.6	6,7
KA4.7	6,4	KB4.7	4,3	KAE4.7	8,1	KBE4.7	5,9
KA4.8	6,5	KB4.8	5,8	KAE4.8	6,5	KBE4.8	5,9
KA4.9	6,2	KB4.9	5,1	KAE4.9	6,5	KBE4.9	6
KA4.10	6,9	KB4.10	4,7	KAE4.10	8,6	KBE4.10	6
KA4.11	6,6	KB4.11	4,8	KAE4.11	8,8	KBE4.11	5,7
KA4.12	8,8	KB4.12	4,9	KAE4.12	7,3	KBE4.12	6,3
KA5.1	8	KB5.1	4,4	KAE5.1	6,5	KBE5.1	5,4
KA5.2	6,5	KB5.2	4,6	KAE5.2	6,7	KBE5.2	5,6
KA5.3	7,4	KB5.3	4	KAE5.3	7,7	KBE5.3	5,6
KA5.4	7,1	KB5.4	5,3	KAE5.4	6,4	KBE5.4	6,3
KA5.5	7,1	KB5.5	4	KAE5.5	6,1	KBE5.5	5,5
KA5.6	6,2	KB5.6	4,2	KAE5.6	6	KBE5.6	5,7
KA5.7	5,6	KB5.7	4,6	KAE5.7	6,8	KBE5.7	5,3
KA5.8	6,3	KB5.8	4	KAE5.8	6,1	KBE5.8	6

KA5.9	6,7	KB5.9	5,2	KAE5.9	7	KBE5.9	5,1
KA5.10	6,5	KB5.10	4,3	KAE5.10	8,8	KBE5.10	6
KA5.11	7	KB5.11	4,4	KAE5.11	7,5	KBE5.11	5,2
KA5.12	6,5	KB5.12	5	KAE5.12	8,3	KBE5.12	5,7
KA6.1	6,9	KB6.1	4,1	KAE6.1	6,9	KBE6.1	6,4
KA6.2	7	KB6.2	4,8	KAE6.2	7,2	KBE6.2	6,3
KA6.3	6,5	KB6.3	4,1	KAE6.3	7,7	KBE6.3	6,1
KA6.4	5,9	KB6.4	4,6	KAE6.4	7,9	KBE6.4	6,4
KA6.5	6,4	KB6.5	4,6	KAE6.5	8,8	KBE6.5	5,6
KA6.6	5,9	KB6.6	4,9	KAE6.6	7,8	KBE6.6	6,1
KA6.7	6,2	KB6.7	5,8	KAE6.7	7	KBE6.7	5,6
KA6.8	8	KB6.8	4,6	KAE6.8	7,6	KBE6.8	5,4
KA6.9	6,9	KB6.9	4,4	KAE6.9	6,7	KBE6.9	5,3
KA6.10	6	KB6.10	4,3	KAE6.10	7	KBE6.10	6,7
KA6.11	7,1	KB6.11	5	KAE6.11	6,5	KBE6.11	5,5
KA6.12	6,2	KB6.12	5	KAE6.12	7,1	KBE6.12	6,8
KA7.1	8,4	KB7.1	4	KAE7.1	7,2	KBE7.1	5,8
KA7.2	5,8	KB7.2	4,1	KAE7.2	7,3	KBE7.2	6
KA7.3	5,7	KB7.3	4,5	KAE7.3	7,2	KBE7.3	5,7
KA7.4	6,6	KB7.4	4,2	KAE7.4	7,7	KBE7.4	6
KA7.5	6	KB7.5	5,2	KAE7.5	7,6	KBE7.5	5,3
KA7.6	6,1	KB7.6	7	KAE7.6	7,4	KBE7.6	5,3
KA7.7	7,2	KB7.7	4,1	KAE7.7	6,5	KBE7.7	6,1
KA7.8	6,7	KB7.8	4	KAE7.8	7,7	KBE7.8	5,1
KA7.9	7,3	KB7.9	4,5	KAE7.9	7,7	KBE7.9	5,1
KA7.10	7,9	KB7.10	4	KAE7.10	6,8	KBE7.10	5,6
KA7.11	7,5	KB7.11	5,1	KAE7.11	7	KBE7.11	5,7
KA7.12	8,5	KB7.12	5	KAE7.12	6,5	KBE7.12	5,1
KA8.1	7,1	KB8.1	8,4	KAE8.1	7,4	KBE8.1	5,4
KA8.2	8,4	KB8.2	5,3	KAE8.2	6,9	KBE8.2	5,2
KA8.3	6,8	KB8.3	5,3	KAE8.3	8,2	KBE8.3	6
KA8.4	8,6	KB8.4	4,4	KAE8.4	7,7	KBE8.4	5,7
KA8.5	8,7	KB8.5	4	KAE8.5	6,9	KBE8.5	5,5
KA8.6	7,9	KB8.6	4,1	KAE8.6	7,1	KBE8.6	6
KA8.7	7,1	KB8.7	5,8	KAE8.7	7,4	KBE8.7	5,5
KA8.8	8	KB8.8	7,9	KAE8.8	6,5	KBE8.8	5,6
KA8.9	6,2	KB8.9	5,4	KAE8.9	6,8	KBE8.9	5,1
KA8.10	7,1	KB8.10	7	KAE8.10	7,5	KBE8.10	5,2
KA8.11	5,9	KB8.11	7,3	KAE8.11	6,9	KBE8.11	6
KA8.12	5,8	KB8.12	7	KAE8.12	6,6	KBE8.12	6,4

Knoop Hardness (Ufi Gel Hard)

Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)
UA1.1	6,5	UB1.1	7,5	UAE1.1	10,2	UBE1.1	7,4
UA1.2	4	UB1.2	8,7	UAE1.2	10,6	UBE1.2	8,6
UA1.3	4,1	UB1.3	8,2	UAE1.3	10,3	UBE1.3	8,5
UA1.4	4	UB1.4	9,2	UAE1.4	10,2	UBE1.4	8,9
UA1.5	5,2	UB1.5	8	UAE1.5	10	UBE1.5	8,5
UA1.6	6,1	UB1.6	8,4	UAE1.6	9,5	UBE1.6	7,3
UA1.7	5,4	UB1.7	8	UAE1.7	9,6	UBE1.7	8
UA1.8	6,3	UB1.8	8,1	UAE1.8	9,6	UBE1.8	6,9
UA1.9	8,6	UB1.9	9,2	UAE1.9	10,2	UBE1.9	7,5
UA1.10	8,6	UB1.10	8,8	UAE1.10	10,5	UBE1.10	8,7
UA1.11	7	UB1.11	9,4	UAE1.11	9,1	UBE1.11	7,9
UA1.12	7,9	UB1.12	7,5	UAE1.12	9,5	UBE1.12	7,5
UA2.1	6,6	UB2.1	7,9	UAE2.1	8,6	UBE2.1	8,4
UA2.2	6,9	UB2.2	8,7	UAE2.2	8,7	UBE2.2	8,4
UA2.3	7,9	UB2.3	9,5	UAE2.3	8,8	UBE2.3	8,2
UA2.4	8,6	UB2.4	9,4	UAE2.4	8,7	UBE2.4	7,5

UA2.5	6	UB2.5	8,1	UAE2.5	9	UBE2.5	8,3
UA2.6	7,1	UB2.6	7,9	UAE2.6	9,3	UBE2.6	7,5
UA2.7	9,8	UB2.7	8,6	UAE2.7	8,9	UBE2.7	8,1
UA2.8	7,8	UB2.8	8	UAE2.8	11	UBE2.8	8,7
UA2.9	8,1	UB2.9	7,3	UAE2.9	10,2	UBE2.9	7,9
UA2.10	9,5	UB2.10	8,3	UAE2.10	10	UBE2.10	8,4
UA2.11	5,1	UB2.11	8,6	UAE2.11	8	UBE2.11	7,7
UA2.12	8,5	UB2.12	8,5	UAE2.12	9,4	UBE2.12	7,9
UA3.1	7,9	UB3.1	7,3	UAE3.1	9,4	UBE3.1	7,9
UA3.2	7,4	UB3.2	8,1	UAE3.2	11,4	UBE3.2	7,8
UA3.3	7,8	UB3.3	7,9	UAE3.3	10,1	UBE3.3	8,1
UA3.4	6,9	UB3.4	9	UAE3.4	9,5	UBE3.4	8,3
UA3.5	6,7	UB3.5	9,1	UAE3.5	9,1	UBE3.5	8,3
UA3.6	7,2	UB3.6	9,2	UAE3.6	9,4	UBE3.6	8,7
UA3.7	5,6	UB3.7	8,7	UAE3.7	9,1	UBE3.7	8,6
UA3.8	7,6	UB3.8	8,8	UAE3.8	9,2	UBE3.8	8,7
UA3.9	6,6	UB3.9	9,2	UAE3.9	9,4	UBE3.9	8,6
UA3.10	6,3	UB3.10	8,7	UAE3.10	8,1	UBE3.10	7,1
UA3.11	6,6	UB3.11	9,1	UAE3.11	10,4	UBE3.11	8,1
UA3.12	6,6	UB3.12	8,2	UAE3.12	9	UBE3.12	8,3
UA4.1	7,3	UB4.1	7,5	UAE4.1	8,8	UBE4.1	8,6
UA4.2	6,8	UB4.2	8,1	UAE4.2	11,1	UBE4.2	7,7
UA4.3	7,9	UB4.3	9	UAE4.3	9,2	UBE4.3	8,7
UA4.4	6,5	UB4.4	7,5	UAE4.4	11,1	UBE4.4	8,5
UA4.5	7,2	UB4.5	8,2	UAE4.5	10,6	UBE4.5	8,2
UA4.6	7	UB4.6	8,5	UAE4.6	10,3	UBE4.6	8,5
UA4.7	7,2	UB4.7	8,1	UAE4.7	10,3	UBE4.7	9
UA4.8	7,6	UB4.8	7,2	UAE4.8	9,1	UBE4.8	8,8
UA4.9	5,9	UB4.9	7,8	UAE4.9	9,9	UBE4.9	7,7
UA4.10	7	UB4.10	7,8	UAE4.10	10,3	UBE4.10	8,2
UA4.11	5,9	UB4.11	8	UAE4.11	9,1	UBE4.11	8,8
UA4.12	6,8	UB4.12	7,4	UAE4.12	10,3	UBE4.12	8,4
UA5.1	6,8	UB5.1	8,3	UAE5.1	10,3	UBE5.1	7
UA5.2	7,3	UB5.2	8,1	UAE5.2	8,8	UBE5.2	7,3
UA5.3	6,9	UB5.3	8,8	UAE5.3	11,3	UBE5.3	8,8
UA5.4	6,2	UB5.4	9,4	UAE5.4	8,5	UBE5.4	7,4
UA5.5	7,4	UB5.5	8,6	UAE5.5	8,8	UBE5.5	7,2
UA5.6	6,1	UB5.6	8,4	UAE5.6	8,1	UBE5.6	7,7
UA5.7	7,3	UB5.7	7,7	UAE5.7	9,5	UBE5.7	7,4
UA5.8	6,6	UB5.8	7,7	UAE5.8	9,3	UBE5.8	8,7
UA5.9	5,9	UB5.9	7,5	UAE5.9	9,2	UBE5.9	8,4
UA5.10	7,4	UB5.10	8	UAE5.10	9,6	UBE5.10	8
UA5.11	8,7	UB5.11	8,2	UAE5.11	9,9	UBE5.11	7,5
UA5.12	9,2	UB5.12	8,8	UAE5.12	9,7	UBE5.12	8,6
UA6.1	7,4	UB6.1	7,7	UAE6.1	8,1	UBE6.1	8,2
UA6.2	7,8	UB6.2	8,2	UAE6.2	9	UBE6.2	8,7
UA6.3	5,6	UB6.3	9,1	UAE6.3	8	UBE6.3	7,2
UA6.4	8,4	UB6.4	7,7	UAE6.4	9,5	UBE6.4	7,6
UA6.5	9,2	UB6.5	8,7	UAE6.5	10,1	UBE6.5	7,9
UA6.6	9,2	UB6.6	7,8	UAE6.6	9,7	UBE6.6	8,3
UA6.7	6,4	UB6.7	7,5	UAE6.7	10,8	UBE6.7	7,4
UA6.8	7	UB6.8	8,1	UAE6.8	7,7	UBE6.8	8
UA6.9	6,6	UB6.9	8,9	UAE6.9	7,8	UBE6.9	8,7
UA6.10	7,4	UB6.10	8,6	UAE6.10	8,6	UBE6.10	8,1
UA6.11	7,5	UB6.11	9	UAE6.11	8	UBE6.11	8,8
UA6.12	7,9	UB6.12	8,4	UAE6.12	9	UBE6.12	8
UA7.1	8,1	UB7.1	8,7	UAE7.1	8,9	UBE7.1	8,3
UA7.2	7,2	UB7.2	7,9	UAE7.2	10	UBE7.2	7,2
UA7.3	6,6	UB7.3	7,7	UAE7.3	8,8	UBE7.3	8,2
UA7.4	9	UB7.4	7,5	UAE7.4	9,6	UBE7.4	8,5
UA7.5	8,2	UB7.5	9,2	UAE7.5	9,1	UBE7.5	8,4
UA7.6	7,1	UB7.6	9,2	UAE7.6	8,6	UBE7.6	7,3
UA7.7	9,2	UB7.7	8,5	UAE7.7	8,4	UBE7.7	7,6
UA7.8	7,5	UB7.8	9,2	UAE7.8	10,3	UBE7.8	7,8

UA7.9	6,5	UB7.9	7,9	UAE7.9	8,8	UBE7.9	8,4
UA7.10	6,7	UB7.10	9	UAE7.10	10,6	UBE7.10	8,7
UA7.11	7,3	UB7.11	8,4	UAE7.11	8,2	UBE7.11	7,5
UA7.12	6,8	UB7.12	8,3	UAE7.12	10,7	UBE7.12	8,1
UA8.1	6	UB8.1	8,1	UAE8.1	9,1	UBE8.1	8,1
UA8.2	10,9	UB8.2	7,8	UAE8.2	8	UBE8.2	7,5
UA8.3	11,2	UB8.3	8,3	UAE8.3	9,7	UBE8.3	7,3
UA8.4	10,1	UB8.4	7,9	UAE8.4	8,3	UBE8.4	7,5
UA8.5	11,5	UB8.5	8,4	UAE8.5	9,9	UBE8.5	7,3
UA8.6	10,1	UB8.6	7,9	UAE8.6	8,3	UBE8.6	8,3
UA8.7	8,3	UB8.7	8,2	UAE8.7	9,6	UBE8.7	7,9
UA8.8	8	UB8.8	8,5	UAE8.8	8,7	UBE8.8	7,9
UA8.9	6,2	UB8.9	9,5	UAE8.9	7,9	UBE8.9	7,8
UA8.10	8	UB8.10	8,5	UAE8.10	7,7	UBE8.10	7,2
UA8.11	11	UB8.11	8,5	UAE8.11	9,1	UBE8.11	7,6
UA8.12	6,5	UB8.12	7,8	UAE8.12	7,9	UBE8.12	8,4

Knoop Hardness (Probase Cold)

Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)	Indentation	Knoop Hardness (KHN)
PCA1.1	12,3	PCB1.1	8,6	PCAE1.1	12	PCBE1.1	9,4
PCA1.2	11	PCB1.2	9,9	PCAE1.2	11,7	PCBE1.2	10,3
PCA1.3	11	PCB1.3	11,8	PCAE1.3	12,9	PCBE1.3	10,1
PCA1.4	11,1	PCB1.4	10,8	PCAE1.4	10,6	PCBE1.4	12
PCA1.5	9,2	PCB1.5	11,1	PCAE1.5	11,8	PCBE1.5	12
PCA1.6	10,7	PCB1.6	10,9	PCAE1.6	12,3	PCBE1.6	11,5
PCA1.7	10,5	PCB1.7	11,6	PCAE1.7	14,1	PCBE1.7	12,3
PCA1.8	9,9	PCB1.8	12	PCAE1.8	13,3	PCBE1.8	13,1
PCA1.9	10,4	PCB1.9	9,9	PCAE1.9	12,1	PCBE1.9	12,4
PCA1.10	12,9	PCB1.10	11,5	PCAE1.10	14,6	PCBE1.10	11,6
PCA1.11	13	PCB1.11	12,6	PCAE1.11	12,8	PCBE1.11	11,4
PCA1.12	12	PCB1.12	11,8	PCAE1.12	12,5	PCBE1.12	9,5
PCA2.1	12,9	PCB2.1	9,6	PCAE2.1	14,3	PCBE2.1	12,7
PCA2.2	14	PCB2.2	12,5	PCAE2.2	11,1	PCBE2.2	13,1
PCA2.3	12,5	PCB2.3	11,3	PCAE2.3	12,3	PCBE2.3	11,5
PCA2.4	14,7	PCB2.4	11,7	PCAE2.4	12,9	PCBE2.4	11,9
PCA2.5	10,5	PCB2.5	12,3	PCAE2.5	12,5	PCBE2.5	11,7
PCA2.6	16,2	PCB2.6	11,4	PCAE2.6	12,3	PCBE2.6	12
PCA2.7	11	PCB2.7	12,5	PCAE2.7	12,9	PCBE2.7	11
PCA2.8	12,7	PCB2.8	12,7	PCAE2.8	12,1	PCBE2.8	11,1
PCA2.9	14,4	PCB2.9	12,4	PCAE2.9	13,3	PCBE2.9	12,7
PCA2.10	9,2	PCB2.10	12,6	PCAE2.10	13,7	PCBE2.10	13,1
PCA2.11	20,8	PCB2.11	12,7	PCAE2.11	14,5	PCBE2.11	11,7
PCA2.12	15,4	PCB2.12	12,3	PCAE2.12	12,7	PCBE2.12	12,4
PCA3.1	12,9	PCB3.1	11,5	PCAE3.1	12,1	PCBE3.1	11,8
PCA3.2	11,1	PCB3.2	11,5	PCAE3.2	11,8	PCBE3.2	12,4
PCA3.3	11,9	PCB3.3	12	PCAE3.3	11,8	PCBE3.3	12,7
PCA3.4	13,5	PCB3.4	11,5	PCAE3.4	12,5	PCBE3.4	12,3
PCA3.5	14,2	PCB3.5	11,6	PCAE3.5	12	PCBE3.5	13,7
PCA3.6	12,3	PCB3.6	11,7	PCAE3.6	12,7	PCBE3.6	10,8
PCA3.7	13,9	PCB3.7	11,4	PCAE3.7	13,8	PCBE3.7	11,1
PCA3.8	10,8	PCB3.8	11,3	PCAE3.8	13,2	PCBE3.8	13,3
PCA3.9	11,4	PCB3.9	11,3	PCAE3.9	9,8	PCBE3.9	12,3
PCA3.10	10,5	PCB3.10	11,8	PCAE3.10	12,1	PCBE3.10	9,9
PCA3.11	13,4	PCB3.11	10,8	PCAE3.11	11,9	PCBE3.11	12,4
PCA3.12	11,8	PCB3.12	11,6	PCAE3.12	10,6	PCBE3.12	13,6
PCA4.1	9,9	PCB4.1	11	PCAE4.1	14,2	PCBE4.1	11,3
PCA4.2	9,7	PCB4.2	12,1	PCAE4.2	12,5	PCBE4.2	12,1
PCA4.3	10,5	PCB4.3	12	PCAE4.3	11,6	PCBE4.3	10,9
PCA4.4	12,4	PCB4.4	11,7	PCAE4.4	13,2	PCBE4.4	11,6

PCA4.5	10,7	PCB4.5	12,3	PCAE4.5	12,7	PCBE4.5	11,9
PCA4.6	11,1	PCB4.6	10,6	PCAE4.6	13,3	PCBE4.6	11
PCA4.7	12,3	PCB4.7	10,6	PCAE4.7	14,4	PCBE4.7	12,3
PCA4.8	12	PCB4.8	10,8	PCAE4.8	15,1	PCBE4.8	10,2
PCA4.9	13,9	PCB4.9	11,5	PCAE4.9	12,6	PCBE4.9	10,9
PCA4.10	11,7	PCB4.10	11,1	PCAE4.10	13,1	PCBE4.10	13,1
PCA4.11	12,3	PCB4.11	12,1	PCAE4.11	11,8	PCBE4.11	12,1
PCA4.12	11,5	PCB4.12	11,3	PCAE4.12	14	PCBE4.12	13,1
PCA5.1	13,6	PCB5.1	10,3	PCAE5.1	12,9	PCBE5.1	11,4
PCA5.2	10,9	PCB5.2	11,2	PCAE5.2	10,6	PCBE5.2	11,2
PCA5.3	13,3	PCB5.3	12,2	PCAE5.3	12,4	PCBE5.3	10,8
PCA5.4	15,2	PCB5.4	10,6	PCAE5.4	12,9	PCBE5.4	10,8
PCA5.5	12	PCB5.5	11	PCAE5.5	13,4	PCBE5.5	12,3
PCA5.6	10,7	PCB5.6	11,4	PCAE5.6	11,3	PCBE5.6	12,8
PCA5.7	9,4	PCB5.7	11,9	PCAE5.7	12,7	PCBE5.7	11,6
PCA5.8	25,3	PCB5.8	11,4	PCAE5.8	11,7	PCBE5.8	11,9
PCA5.9	5,8	PCB5.9	11,3	PCAE5.9	11,6	PCBE5.9	11,5
PCA5.10	18,2	PCB5.10	10,4	PCAE5.10	12,5	PCBE5.10	10,9
PCA5.11	12,2	PCB5.11	12,1	PCAE5.11	12	PCBE5.11	11,5
PCA5.12	10,5	PCB5.12	10,9	PCAE5.12	12,2	PCBE5.12	11,8
PCA6.1	11,7	PCB6.1	10,6	PCAE6.1	14	PCBE6.1	12,2
PCA6.2	15	PCB6.2	10,9	PCAE6.2	14,7	PCBE6.2	12,4
PCA6.3	9,2	PCB6.3	10,7	PCAE6.3	12,3	PCBE6.3	12,5
PCA6.4	11,7	PCB6.4	11,1	PCAE6.4	13,1	PCBE6.4	9,9
PCA6.5	11,1	PCB6.5	10,9	PCAE6.5	12,7	PCBE6.5	11,2
PCA6.6	6,2	PCB6.6	10,8	PCAE6.6	13,6	PCBE6.6	11,7
PCA6.7	10,3	PCB6.7	11,9	PCAE6.7	14,1	PCBE6.7	12,5
PCA6.8	11,4	PCB6.8	10,9	PCAE6.8	13,5	PCBE6.8	11,8
PCA6.9	11,1	PCB6.9	10,3	PCAE6.9	12,8	PCBE6.9	11,5
PCA6.10	10,1	PCB6.10	10,2	PCAE6.10	11,1	PCBE6.10	13,7
PCA6.11	12,4	PCB6.11	10,6	PCAE6.11	11,4	PCBE6.11	10,8
PCA6.12	10,3	PCB6.12	12,1	PCAE6.12	12	PCBE6.12	10,6
PCA7.1	12,3	PCB7.1	11	PCAE7.1	11	PCBE7.1	10,1
PCA7.2	12	PCB7.2	11,4	PCAE7.2	10,6	PCBE7.2	10,1
PCA7.3	11,1	PCB7.3	11,4	PCAE7.3	10,9	PCBE7.3	12,9
PCA7.4	12,3	PCB7.4	11,1	PCAE7.4	13,1	PCBE7.4	11,9
PCA7.5	10,3	PCB7.5	10	PCAE7.5	12,7	PCBE7.5	12,7
PCA7.6	12,1	PCB7.6	11,4	PCAE7.6	11,4	PCBE7.6	12,9
PCA7.7	9,5	PCB7.7	11,3	PCAE7.7	14	PCBE7.7	11,8
PCA7.8	10,1	PCB7.8	11,3	PCAE7.8	11,2	PCBE7.8	11,2
PCA7.9	11,3	PCB7.9	10	PCAE7.9	13,5	PCBE7.9	13,1
PCA7.10	12,7	PCB7.10	10,8	PCAE7.10	12,2	PCBE7.10	9,7
PCA7.11	11,8	PCB7.11	10,5	PCAE7.11	13	PCBE7.11	12,2
PCA7.12	10,5	PCB7.12	11,3	PCAE7.12	13,6	PCBE7.12	11,2
PCA8.1	16	PCB8.1	12,3	PCAE8.1	8,8	PCBE8.1	12,3
PCA8.2	13,6	PCB8.2	12,1	PCAE8.2	10,8	PCBE8.2	11,3
PCA8.3	11,5	PCB8.3	11,7	PCAE8.3	12,5	PCBE8.3	10,8
PCA8.4	12,2	PCB8.4	12,3	PCAE8.4	13,5	PCBE8.4	10,8
PCA8.5	9,9	PCB8.5	12,6	PCAE8.5	12,9	PCBE8.5	12,1
PCA8.6	14,1	PCB8.6	11,4	PCAE8.6	11,1	PCBE8.6	10,8
PCA8.7	13,6	PCB8.7	11,6	PCAE8.7	9,3	PCBE8.7	11,2
PCA8.8	11	PCB8.8	10,6	PCAE8.8	13,9	PCBE8.8	9,7
PCA8.9	14,7	PCB8.9	12,5	PCAE8.9	8,4	PCBE8.9	10,9
PCA8.10	12,9	PCB8.10	12	PCAE8.10	10,6	PCBE8.10	11,5
PCA8.11	11,9	PCB8.11	12,1	PCAE8.11	11,7	PCBE8.11	11,5
PCA8.12	10,9	PCB8.12	10,6	PCAE8.12	9,4	PCBE8.12	10,8

Flexural Strength (Kooliner)

Specimen	Load at Yield (kN)	Stress at Yield (MPa)	Modulus (MPa)	Width (mm)	Thickness (mm)	Flexural Strength (MPa)
KA1	0,0622	45,17	1156	10,34	3,16	45,18
KA2	0,0637	48,76	1171	10,06	3,12	48,79
KA3	0,0687	49,12	1129	10,11	3,22	49,15
KA4	0,0694	48,11	1195	10,37	3,23	48,11
KA5	0,0611	44,4	1289	10,15	3,19	44,37
KA6	0,0711	46,61	1213	10,51	3,3	46,59
KA7	0,0552	43,77	1375	10,04	3,07	43,75
KA8	0,0643	47,57	1290	10,21	3,15	47,60
KB1	0,0576	43,31	1200	10,05	3,15	43,32
KB2	0,0571	42,51	1153	10,03	3,17	42,49
KB3	0,0468	33,53	1056	10,36	3,18	33,50
KB4	0,0534	35,18	1179	10,45	3,3	35,19
KB5	0,0468	35,59	1147	10,14	3,12	35,56
KB6	0,0449	32,65	1152	10,02	3,21	32,62
KB7	0,0541	41,19	1175	10,13	3,12	41,15
KB8	0,0467	33,06	1232	10,22	3,22	33,05
KAE1	0,0629	45,28	1399	10,05	3,22	45,27
KAE2	0,0676	47,62	1267	10,15	3,24	47,58
KAE3	0,059	45,6	1378	10,03	3,11	45,61
KAE4	0,0642	47,49	1292	10,03	3,18	47,47
KAE5	0,0583	41,78	1283	9,97	3,24	41,78
KAE6	0,0635	44,3	1353	10,12	3,26	44,28
KAE7	0,0554	40,54	1280	10,01	3,2	40,54
KAE8	0,0599	45,05	1332	9,98	3,16	45,08
KBE1	0,0481	34,34	1235	10,06	3,23	34,37
KBE2	0,0445	34,5	976,2	10,06	3,1	34,52
KBE3	0,049	35,75	1157	10,04	3,2	35,75
KBE4	0,0477	35,78	1031	10,01	3,16	35,79
KBE5	0,0499	35,92	1101	10,05	3,22	35,92
KBE6	0,0533	37,05	1041	10,15	3,26	37,06
KBE7	0,0432	32,64	1212	10,12	3,13	32,68
KBE8	0,048	36,46	1046	9,96	3,15	36,43

Flexural Strength (Ufi Gel Hard)

Specimen	Load at Yield (kN)	Stress at Yield (MPa)	Modulus (MPa)	Width (mm)	Thickness (mm)	Flexural Strength (MPa)
UA1	0,0641	53,39	1992	9,95	3,14	49,00
UA2	0,0635	52,95	2171	10,22	3,22	44,94
UA3	0,0698	58,17	2175	10,12	3,22	49,89
UA4	0,0426	35,49	2035	10,25	3,13	31,82
UA5	0,0792	66,01	2233	10,39	3,29	52,82
UA6	0,0409	34,12	2099	10,21	3,15	30,28
UA7	0,0589	49,11	2203	10	3,24	42,08
UA8	0,0298	24,87	2318	10,21	3,28	20,35
UB1	0,0516	38,56	1778	10,11	3,15	38,58
UB2	0,0574	43,99	1814	9,93	3,14	43,97
UB3	0,0516	41,47	1782	9,9	3,07	41,48
UB4	0,0421	31,83	1727	10,12	3,13	31,85
UB5	0,0503	38,47	1769	9,89	3,15	38,44
UB6	0,038	28,53	1823	9,99	3,16	28,57
UB7	0,0523	40,38	1756	9,98	3,12	40,38
UB8	0,0432	33,12	1811	9,85	3,15	33,15
UAE1	0,0626	47,06	1780	10,12	3,14	47,05
UAE2	0,0629	47,69	1726	9,97	3,15	47,69
UAE3	0,0517	39,75	1693	10,15	3,1	39,75

UAE4	0,0764	56,88	1603	10,09	3,16	56,87
UAE5	0,053	41,62	1787	10,06	3,08	41,65
UAE6	0,056	42,7	1771	9,97	3,14	42,73
UAE7	0,0612	44,72	1623	10,34	3,15	44,74
UAE8	0,0603	46,12	1692	10,07	3,12	46,14
UBE1	0,0487	34,74	1615	10,27	3,2	34,73
UBE2	0,0375	27,63	1696	10,08	3,18	27,59
UBE3	0,05	37,22	1652	9,91	3,19	37,19
UBE4	0,0298	22,36	1681	10,15	3,14	22,33
UBE5	0,0566	40,87	1678	10,27	3,18	40,87
UBE6	0,038	27,57	1556	10,11	3,2	27,53
UBE7	0,0412	31,02	1715	10,05	3,15	30,99
UBE8	0,0434	32,59	1676	10,01	3,16	32,56

Flexural Strength (Probase Cold)

Specimen	Load at Yield (kN)	Stress at Yield (MPa)	Modulus (MPa)	Width (mm)	Thickness (mm)	Flexural Strength (MPa)
PCA1	0,1113	92,75	2660	10,12	3,25	78,09
PCA2	0,1171	97,58	2800	10,27	3,37	75,30
PCA3	0,1123	93,58	2527	10,17	3,26	77,93
PCA4	0,1016	84,67	2235	10,29	3,18	73,23
PCA5	0,1171	97,58	2609	10,42	3,25	79,80
PCA6	0,1177	98,08	2424	9,84	3,23	85,99
PCA7	0,1038	86,5	2187	10,14	3,09	80,41
PCA8	0,1123	93,58	2497	10,39	3,21	78,67
PCB1	0,0791	58,58	2252	10,14	3,16	58,59
PCB2	0,0675	50,25	2281	9,66	3,23	50,23
PCB3	0,0705	49,47	2087	10,25	3,23	49,44
PCB4	0,0843	60,45	2233	9,96	3,24	60,47
PCB5	0,0737	53,3	2070	10,13	3,2	53,29
PCB6	0,0785	55,22	1870	10,03	3,26	55,23
PCB7	0,0784	56,5	2173	10,16	3,2	56,52
PCB8	0,07	53,37	2061	10,17	3,11	53,37
PCAE1	0,1022	77,09	2192	10,02	3,15	77,09
PCAE2	0,127	92,25	2107	10,02	3,21	92,25
PCAE3	0,1059	83,52	1982	9,96	3,09	83,52
PCAE4	0,1058	76,52	2133	10,19	3,19	76,52
PCAE5	0,0883	61,11	2175	10,45	3,22	61,12
PCAE6	0,0892	63,52	2266	9,97	3,25	63,53
PCAE7	0,1094	82,2	2046	10,06	3,15	82,20
PCAE8	0,1067	75,87	1994	10,11	3,23	75,87
PCBE1	0,074	54,81	2194	9,83	3,21	54,79
PCBE2	0,0647	50,61	2166	9,98	3,1	50,60
PCBE3	0,083	62,02	2260	9,68	3,22	62,02
PCBE4	0,0916	67,24	2204	9,86	3,22	67,20
PCBE5	0,0784	58,48	2203	10,2	3,14	58,47
PCBE6	0,0857	63,81	2106	9,96	3,18	63,82
PCBE7	0,0767	60,19	2182	9,95	3,1	60,16
PCBE8	0,0782	57,72	2106	9,99	3,19	57,69

Wettability (Kooliner)

Specimen	Width (mm)	Height (mm)	Thickness (mm)	Advance contact angle (°)		γTotal (mN/m)	γdisperse (mN/m)	γpolar (mN/m)
				Water	1,2-propanediol			
KA1	25,28	15	1,08	94,41	52,87	25,8	17,2	8,6
KA2	25,28	17,88	1,1	91,21	56,95	25,7	14,3	11,3
KA3	25,26	14,5	1,12	97,14	51,21	26	19,3	6,8

KA4	25,12	14,88	1,08	94,75	53,59	25,6	17	8,6
KA5	25,28	15,28	1,08	95,04	56,68	24,7	15,7	9
KA6	25,24	16	1,08	93,26	54,15	25,7	16,2	9,6
KA7	25,28	15,5	1,06	95,31	48,43	27,1	19,7	7,4
KB1	23,44	14,28	1,1	75,09	44,88	34,4	15,2	19,2
KB2	25,38	14,4	1,08	72,13	38,11	36,8	16,8	20
KB3	25,26	13,96	1,08	77,42	44,5	33,3	15,7	17,6
KB4	25,1	14,84	1,08	74,75	36,85	35,7	17,7	18
KB5	24,74	15,18	1,08	73,08	34,43	36,8	18	18,8
KB6	25,5	14,18	1,08	72,48	30,89	37,6	19	18,6
KB7	25,52	14,38	1,08	75,1	31,33	36,4	19,4	17

Wettability (Ufi Gel Hard)

Specimen	Width (mm)	Height (mm)	Thickness (mm)	Advance contact angle (°)		γTotal (mN/m)	γdisperse (mN/m)	γpolar (mN/m)
				Water	1,2-propanediol			
UA1	24,67	17,33	1,14	74,26	39,5	35,5	16,8	18,8
UA2	25,26	18,19	1,14	75,2	40,19	35	16,7	18,3
UA3	24,46	18,05	1,1	74	32,55	36,7	18,8	17,9
UA4	24,36	19,27	1,1	72,99	32,49	37,2	18,6	18,5
UA5	24,49	17,8	1,11	69,81	35,28	38,3	17,2	21,1
UA6	24,56	17,18	1,1	73,25	39,31	36,1	16,6	19,4
UA7	24,63	17,86	1,11	69,8	35,68	38,3	17,1	21,1
UB1	25,63	18,1	1,15	69,84	25,07	39,6	19,9	19,7
UB2	25,63	15,99	1,16	70,3	27,55	39,1	19,4	19,6
UB3	25,46	17,91	1,14	70,83	31,38	38,3	18,5	19,8
UB4	25,64	18,83	1,08	74,81	24,61	37,6	21,1	16,4
UB5	25,27	18,01	1,17	70,72	27,19	38,9	19,6	19,3
UB6	25,21	17,54	1,13	72,06	29,24	38	19,3	18,7
UB7	25,13	17,44	1,16	66,04	31,74	40,7	17,6	23,1

Wettability (Probase Cold)

Specimen	Width (mm)	Height (mm)	Thickness (mm)	Advance contact angle (°)		γTotal (mN/m)	γdisperse (mN/m)	γpolar (mN/m)
				Water	1,2-propanediol			
PCA1	25,36	16,99	1,14	83,28	42,23	31,4	17,9	13,5
PCA2	25,33	19,04	1,12	73,74	42,01	35,5	15,9	19,6
PCA3	25,14	17,16	1,12	69,39	40,82	37,9	15,6	22,3
PCA4	25,55	18,31	1,15	78,17	38,15	34	18	16
PCA5	25,47	18,22	1,15	76,39	41,07	34,3	16,7	17,7
PCA6	25,75	18,94	1,11	72,67	42,63	35,9	15,5	20,4
PCA7	25,06	17,6	1,02	75,01	30,84	36,54	19,52	17,01
PCB1	25,55	18,41	1,1	71,09	36,29	37,5	17,2	20,4
PCB2	25,75	17,3	1,13	71,05	35,49	37,7	17,4	20,3
PCB3	25,47	17,93	1,15	75,37	34,88	35,7	18,4	17,3
PCB4	25,4	17,65	1,14	67,95	34,58	39,4	17,1	22,2
PCB5	25,85	18,66	1,12	68,33	28,54	39,9	18,8	21,1
PCB6	25,5	18,64	1,1	60,85	28,24	43,9	17,8	26,1
PCB7	25,5	18,72	1,08	68,93	29,44	39,5	18,7	20,8