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Dynamic mechanical properties of oral mucosa: Comparison with polymeric soft denture liners

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ABSTRACT

The purpose of this work was to characterize the viscoelastic behaviour of oral mucosa and compare it with the dynamic mechanical properties of different soft liners. For this purpose, a sample of pig oral mucosa and six commercialized soft liner samples have been investigated. A comparison was also carried with the first suitable hard rubber for dental prosthetics: vulcanite. Creep recovery (CR) and dynamic mechanical analysis (DMA) have been used to determine the mechanical modulus of oral mucosa and soft liners respectively. The Poisson ratio is used to compare mucosa bulk modulus and soft liner shear modulus. The biomechanical concept of conventional complete dentures needs a good adjustment of dynamic mechanical impedance between the base and oral mucosa. The viscoelastic mechanical property of the oral mucosa as a referent biopolymer has been confirmed in vitro. The modulus value, adjusted for old patients in physiological conditions, is in the order of 3 MPa. This study underlines the plasticization effect of absorbed water on the mechanical properties of the underlying tissue. This study allows us to define some characteristics of the most adapted biomaterial according to the clinical exigency. The required biomaterial must display the following properties: compatibility and chemical resistance with biological environment perpetuated mechanical properties during physiological conditions and clinical use, good adjustment of dynamic mechanical impedance with supporting mucosa and easy sample processing.

1. Introduction

Edentulous people wearing removable complete dentures, and especially old people present frequently painful oral mucosa. These patients are uncomfortable with their complete dentures and even they cannot wear them. The underlying oral mucosa is usually compressed, indeed sheared between the bone ridge and the acrylic hard base. A prolonged and close contact between the edentulous oral mucosa and the inner surface of the complete denture, associated with the transmission of occlusal forces involve tissue diseases, bone resorption and pain in the short and long term.

To overcome these difficulties and improve the biological integration, the setting of a polymeric soft liner onto the

hard denture base allows a limited transmission of the forces and pressures generated during mastication and may lead to a great relief of oral mucosa pain. Soft liners produce a cushioning effect and so constitute an attractive medical response for elderly patients (Braden et al., 1995; Rueggeberg, 2002; Wright, 1994, 1984; Jagger, 1997).

For a long time, dentists have used several kinds of polymeric soft lining materials such as natural rubbers, plasticized acrylic or more recently silicone elastomers (Wright, 1984; Jagger, 1997; Wright, 1981). These soft lining materials have very different mechanical properties. But, their longevity and durability are still a problem: ageing affects the mechanical properties or increases tear (Wright, 1981; Tamura et al., 2002; Wright, 1976; McCabe et al., 2002, 1996; Buch and Beal, 1995; Murata et al., 1998b; Waters and Jagger, 1999; Braden et al., 1997).

The purpose of this study was to characterize the viscoelastic behaviour of oral mucosa and compare it to the dynamic mechanical properties of different soft liners. A sample of porcine oral mucosa and six commercialized soft liner samples, used widely for their well-known clinical properties, have been investigated. Furthermore, a comparison was carried out with the first suitable hard rubber for dental prosthetics: vulcanite. Creep recovery (CR) and dynamic mechanical analysis (DMA) have been used to determine the mechanical modulus of oral mucosa and soft liners respectively.

2. Experimental part

2.1. Materials

The porcine oral mucosa is similar to the human oral mucosa in terms of structure, permeability and composition. Porcine buccal tissues have been currently used as models for human tissues; because they are omnivorous and oral mucosa is stimulated in the same way during the function (chewing) (Wertz et al., 1993; Squier and Wertz, 1993; Shojaei, 1998; De Caro et al., 2008; Kulkarni and Mahalingam, 2009). Porcine oral mucosa samples were taken on a fresh mandible a few hours after animal slaughter. A mucoperiosteum flap was sampled in full thickness of the vestibular or lingual attached gingival, opposite to the molars. 10 mm-diameter and 1.5 mm-thick disks were cut with gingival scissors to avoid tearing of the tissues. The main feature of the oral cavity environment is the presence of saliva (saliva is 99% water); a first sample was kept in water during the mechanical test (hydrated sample). A second one was dried 30 min before the mechanical test at ambient temperature.

Two acrylic, four silicone and polyisoprene permanent soft liners were investigated. Commercial characteristics of the soft liners are listed in Table 1. Mechanical test samples were obtained using dental stone mould according to the prosthetic technique. A plaster of Paris mould was prepared from a wax model inserted in a muffle. All the soft liners were prepared and cured according to the manufacturer's instructions (Braden et al., 1995; Rueggeberg, 2002; Wright, 1994).

Polyacrylic processing needs to mix powder and liquid: 4 cm^3/6 ml for PMMA, 8 cm^3/1.6 ml for PEMA. The muffle was filled with the mixture under a pressure of 200 bar at ambient temperature. Then the PMMA muffle was placed in a water bath at 70 °C for 3 h, then at 100 °C for 30 min. The PEMA muffle was placed in a water bath at 75 °C for 2 h and then air-cooled.

Polyisoprene sample processing was carried out using four sheets of non-vulcanized polyisoprene, put into a muffle under a pressure of 300 bar. In a saturated steam environment, the muffle was submitted to a thermal cycle where the temperature was raised from 40 to 165 °C at a rate of 4 °C/min, followed by 90 min at 165 °C and then a slow cooling from 165 to 40 °C at 4 °C/min.

Polysiloxanes, namely RTV as room temperature vulcanisation polymerise at ambient temperature. Their processing needs mixing the same amount of two pastes. The muffle was filled with the mixture under a pressure of 300 bar at ambient temperature. The muffle was plunged into a water bath for 20 min at 45 °C. The HTV polysiloxane process requires to fill one paste into the muffle under a pressure of 250 bar. Then the muffle was submitted to a temperature cycle: a slow temperature rising from ambient temperature to 100 °C, followed by 2 h at 100 °C, then air-cooled down to ambient temperature.

After setting, all samples were removed from the moulds in the open air at an ambient temperature (Braden et al., 1995; Rueggeberg, 2002; Wright, 1981; Tamura et al., 2002; Wright, 1976).

2.2. Methods

The mechanical properties of the porcine oral mucosa were investigated using a TMA/DMA 7 Perkin Elmer in static or dynamic solicitation. The compression mode between two parallel plates was used in creep recovery (CR) and dynamic mechanical (DM) tests. In a CR test a compression load of 0.32 kPa was applied for 5 min at constant temperature; the load was suppressed and the deformation decrease was recorded (Stafford et al., 1975).

In a DM experiment hydrated and dried samples were studied at a constant frequency of 1 Hz. The time dependence of the Young Modulus in compression, labelled compression modulus E', was recorded at a constant temperature of 37 °C. Two consecutive tests were done for each sample. These frequency and temperature conditions mimic the cyclic masticatory rhythm and human body temperature (Buch and Beal, 1995; Waters and Jagger, 1999; Murata et al., 1998a, 2000, 2002; Williams et al., 1996).

Dynamic mechanical properties of soft liner samples were carried out using an ARES strain controlled rheometer in the torsion rectangular mode from TA Instruments. ARES test samples were parallelepiped with 44 × 11.5 × 1.5 mm as dimensions in length × width × thickness respectively.

An oscillating strain $\gamma^*(t)$ was applied to the sample and the resulting stress $\sigma^*(t)$ was measured with:

$$\gamma^*(t) = \gamma_0 e^{i\omega t} \quad \text{and} \quad \sigma^* = \sigma_0 e^{i[\omega t + \delta]} \tag{1}$$

where δ is the phase shift angle.

Table 1 – Investigated soft denture liner materials.

Product	Polymer	Generic type	Manufacturer
Vertex®	Poly(methylmethacrylate)	Plasticized PMMA	Dentimex B.V. (Nederland)
Softerex®	Poly(ethylmethacrylate)	Plasticized PEMA	Zhermack (Italy)
Molloplast®	Poly(siloxane)[b]HT	Siloxane HTV	Detax GmbH (Germany)
GC Reline®	Poly(siloxane)[b]RT	Siloxane RTVg	GC dental products (Japan)
Permafix®	Poly(siloxane)[b]RT	Siloxane RTVp	Kohler GmbH (Germany)
Ufigel®	Poly(siloxane)[b]RT[a]	Siloxane RTVu	Voco GmbH (Germany)
Vulcanite®	Poly(isoprene)	Polyisoprene	Laboratoire Delac (France)

[a]HT = Heat cured sample.
[b]RT = Cold cured sample.

The complex modulus $G^*(\omega)$, was determined from the following relation:

$$G^*(\omega) = G'(\omega) + iG''(\omega) = \frac{\sigma_0}{\gamma_0} \exp[i\delta(\omega)] \quad (2)$$

where G' and G'' are the storage (or elastic) and loss modulus respectively.

G' measures the ability of the material to store energy associated with a recoverable elastic deformation. G'' relates to the dissipative, viscous component (Williams et al., 1996; McCrum et al., 1991; Ward, 1971; Saber-Sheikh et al., 1999; Waters et al., 1996; Clarke, 1989a,b). The imposed maximum strain amplitude γ_0 was well within the linear viscoelasticity range, i.e., 0.2% for acrylic, silicone soft liners and 0.01% for polyisoprene soft liners. All experiments were carried out on two samples.

3. Results

The isothermal creep and creep recovery experiments for the hydrated porcine oral mucosa have been reported in Fig. 1. At 37 °C, data obtained for the hydrated sample were shown. The creep recovery test has been obtained by sudden release of the applied stress when the sample has undergone a compression creep deformation of 3.2%. An elastic instantaneous strain of 0.01 (1%) has been observed since load was removed. The gradual increase in strain until 5 min after the stress was applied, called the creep strain, is characteristic of the viscoelastic behaviour. For 5 min, the total deformation was increased by 3 times. The creep recovery observed for 30 min was also associated with the viscoelasticity of the oral mucosa. A residual strain of $\gamma_0 = 0.25\%$ was measured corresponding to the irreversible creep.

Fig. 2 shows the temperature dependence of the storage modulus $G'(T)$ for the various soft liners. $G'(T)$ was measured as a function of temperature ranging from −150 to 70 °C with a heating rate of 3 °C min^{-1} and ω = 1 rad s^{-1} as the fixed angular frequency (McCabe et al., 2002; Stafford et al., 1975; Van Krevelen and Hoftyzer, 1972). Viscoelastic behaviour has been observed for all the soft liners as expected. A similar mechanical behaviour was shown for the same polymer family. Two drops in G' which occurred around −120 and −40 °C were shown for polysiloxane samples. These drops were the thermo-mechanical manifestation of the glass–rubber transition, namely the α main mechanical

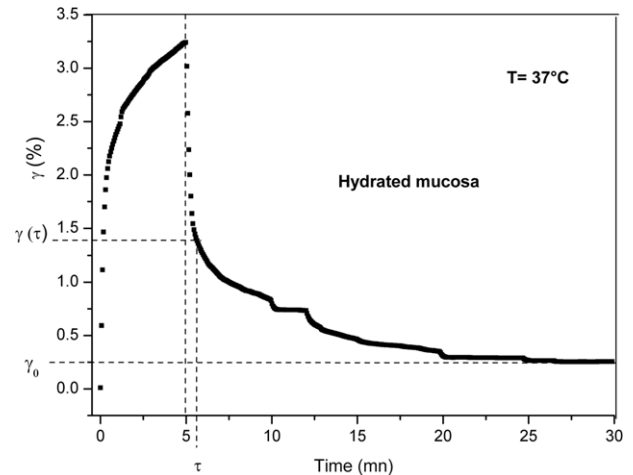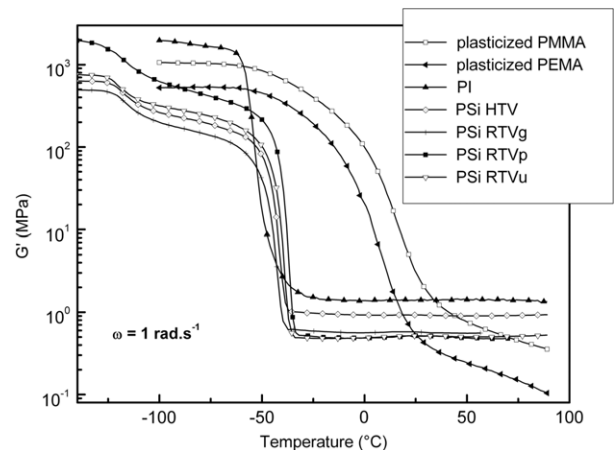**Fig. 1 – Creep recovery of hydrated oral mucosa.**

Fig. 2 – Storage modulus $G'(T, 1$ rad s$^{-1})$ as a function of temperature at the angular frequency of 1 rad s^{-1} for all the soft liner samples: (□) plasticized PMMA, (◄) plasticized PEMA, (▲) PI, (◇) PSi HTV, (+) PSi RTVg, (■) PSi RTVp, (▽) PSi RTVu.

relaxation, and softening of the polysiloxane permanent soft liner (associated with melting) respectively. In the studied temperature range, only one drop was observed for the PI and linear polymer (PMMA and PEMA) associated with mechanical manifestation of the glass transition.
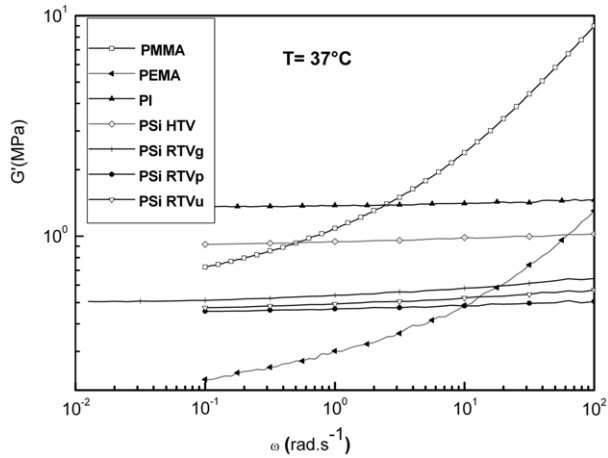
Fig. 3 – Storage modulus $G'(\omega)$ as a function of frequency taken at the mucosa temperature of 37 °C for all the soft liner samples: (□) plasticized PMMA, (◄) plasticized PEMA, (▲) PI, (◇) PSi HTV, (+) PSi RTVg, (■) PSi RTVp, (▽) PSi RTVu.

Fig. 5 – Storage modulus G' (t, 1 rad s^{-1}, 37 °C) as a function of time at the angular frequency of 1 rad s^{-1}, at the temperature of 37 °C for all the soft liner samples: (□) plasticized PMMA, (◄) plasticized PEMA, (▲) PI, (◇) PSi HTV, (+) PSi RTVg, (■) PSi RTVp, (▽) PSi RTVu.

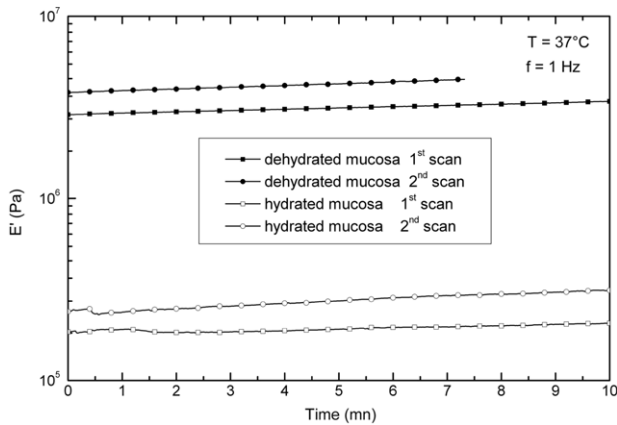**Fig. 4 – Compression modulus E' (t, 1 rad s^{-1}, 37 °C) as a function of time at the angular frequency of 1 rad s^{-1}, at the temperature of 37 °C for oral mucosa samples.**

The storage modulus $G'_T(\omega)$ has been plotted as a function of the frequency in Fig. 3. Experiments were carried out at 37 °C, for a frequency sweep from 10^{-1} to 100 rad s^{-1} (Mesquita et al., 2006). For PMMA and PEMA, $G'_T(\omega)$ increased with frequency, and as expected from the temperature dependence of G' (see Fig. 2) this increase was drastic. Polysiloxane and polyisoprene moduli remained constant along the frequency run.

Fig. 4 shows the time dependence of compression modulus with oral mucosa hydration. In order to be in agreement with the oral and occlusal conditions, E' was measured along 20 min at 37 °C and $\omega = 1$ rad s^{-1}. The compression modulus decreased with absorption of water. Two runs were carried out; stiffening of the oral mucosa was observed for the second compression tests.

Fig. 5 shows that in physiological conditions, PI and PMMA exhibited a high value of the modulus while PEMA a lower
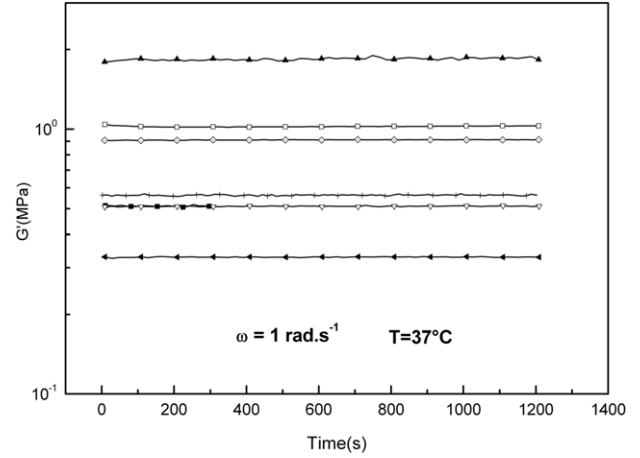
Table 2 – Comparison of strains for mucosa samples *in vivo* and *in vitro*.

	Mucosa *In vivo* (Clarke, 1989b; Jerolimov et al., 1991)	Mucosa *In vitro*
Instantaneous strain	67%	46%
Retarded strain	16.5%	46%
Permanent strain	16.5%	8%

one. All the RTV polysiloxanes have the same G' while the HTV polysiloxane displayed a higher value.

4. Discussion

Inoue et al. (1985), Kydd et al. (1971) and Kydd and Daly (1982) have investigated viscoelastic behaviour of dog and human oral mucosa in vivo by the creep recovery test. In their test, a compression load was applied at a frequency of 10 kHz for 10 min. They assumed that this high frequency test mimics the masticatory forces applied in vivo and transmitted to the underlying tissues. The different contribution to the total strain are reported in Table 2 and compared to the values obtained by Kydd et al. 's Porcine oral mucosa tested in vitro has a best creep recovery than oral mucosa in vivo. The value of relaxation time is a few minutes, close to the value reported by Kydd et al.. The value of 2.72 MPa is found for the compression modulus of the oral mucosa (Inoue et al., 1985).

The physical structure of polysiloxanes consists of two components: one of them shows a random amorphous and the other a regular structure even in the molten state (Takahashi et al., 2001). This last regular structure can be considered as a crystalline phase. As expected for silicone soft liners, the G' value is independent of temperature above -25 °C. The curve associated with polyisoprene is characteristic of an entangled polymer like

polysiloxanes. But PI exhibits a greater modulus and its glass transition temperature occurs in the same temperature range as the melting of the crystalline phase of polysiloxanes. Acrylics soft liner behaviour is characteristic of a linear polymer. In the investigated temperature ranging from −50 to 25 °C, the large decrease of storage modulus is associated with the glass–rubber transition. The glass transition temperature of PMMA is 130 °C (Clarke, 1989a,b). The chemical formulation of the PMMA sample (Vertex), according to the manufacturer, includes aromatic esters acting as plasticizers. The plasticization of PMMA chains explains the drastic decrease of 110 °C in T_g value. The same remark is valid for the PEMA (Softerex) sample: the glass transition temperature is also drastically lower than the well-known value in pure PEMA, i.e. 85 °C (Clarke, 1989a,b).

The detailed chemical composition of Softerex is unknown. PEMA is the major component. The plasticization of acrylic soft liners confers to the samples a rubbery behaviour at the physiological temperature of 37 °C as expected for clinical use. Above 30 °C, the polymer chains flow without marked rubbery plateau as expected in a non-entangled polymer. The low molecular weight of PMMA and PEMA samples explain this characteristic rheological behaviour. Around the oral mucosa temperature, the storage shear modulus of acrylic soft liners is a slowly decreasing function of the temperature.

Hydration is crucial for all biologic tissues since it strongly modifies their mechanical properties. Clinically, a loss of hydration results in an increase of rigidity. Such behaviour is found with polyamide probably due to the analogy of amide functional groups (Stafford et al., 1986; Mc Gregor et al., 1984). Mechanical modulus increases with hydration of oral mucosa (Inoue et al., 1985; Kydd et al., 1971; Kydd and Daly, 1982; Sloan et al., 1991). Mechanical modulus of soft liners is determined from the well known relation:

$$E = 3K(1 − 2\nu) = 2G(1 + \nu)$$

where E is the Young modulus, K the bulk modulus and ν the Poisson ratio (ν is equal to 0.33 for amorphous polymers, 0.4 for crystalline polymers and 0.49 for elastomer (Van Krevelen and Hoftyzer, 1972)). The medium is assumed to be isotropic.

K values are reported in Table 3. The dehydrated oral mucosa modulus is proposed as the reference value. In geriatrics, it is the most representative of the clinical reality (Inoue et al., 1985; Kydd et al., 1971; Kydd and Daly, 1982; Sloan et al., 1991). Compression modulus value of soft liners calculated from G' is in agreement with the value measured for the oral mucosa. At 37 °C, PMMA modulus has the same value as oral mucosa whereas PEMA displays a lower value. For all the acrylic soft liners, their mechanical properties are modified by temperature and frequency. Variable modulus and non-reticulated structure are responsible for the decrease of their mechanical properties and their fast ageing.

The high value of the polyisoprene modulus recovers the same range of the oral mucosa. We recall that polyisoprene was used fifty years ago as the entire denture base. Its physical structure explains its excellent mechanical properties. Polyisoprene was dropped due to its difficult processing and poor cosmetic quality. HTV polysiloxane displays the same bulk modulus value as polyisoprene. RTV

Table 3 – Different values of moduli at 37 °C (K is a calculated compression modulus, G' is an experimental storage modulus).

Samples	K (MPa)	G' (MPa)
Plasticized PMMA	2.6	1
Plasticized PEMA	0.86	0.33
Polyisoprene	89.4	1.8
Siloxane HTV	45.2	0.91
Siloxane RTVg	27.8	0.56
Siloxane RTVp	25.3	0.51
Siloxane RTVu	25.3	0.51
"dehydrated" mucosa	$E' = 2.72$ MPa	

polysiloxanes have bulk modulus values close to the oral mucosa. Their permanent rubbery behaviour is a guaranty for long use. But the low values of modulus make it inconvenient to use polysiloxane as an entire denture base; they have to be bonded on a hard base. Clinicians know the difficulties of sticking silicone soft liner on an acrylic hard base. However, polysiloxanes are actually the soft liners used more often by dentists: easy for prosthetic making, and preservation of mechanical properties.

5. Conclusion

Conventional complete dentures need a good adjustment of dynamic mechanical impedance between the base and oral mucosa. First, the viscoelastic mechanical property of the oral mucosa as a reference biopolymer has been confirmed in vitro. The compression modulus value, adjusted for old patients in physiological conditions, is in the order of 3 MPa. This study underlines the plasticization effect of absorbed water on the mechanical properties of the underlying tissue. Then, the rheological behaviour of all soft liners is viscoelastic. This study allows us to define the characteristics of the "perfect" biomaterial (the most adapted according to clinical exigency). The required biomaterial must display the following properties: compatibility and chemical resistance with biological environment, durability of mechanical properties during physiological conditions and clinical use, good adjustment of dynamic mechanical impedance with supporting mucosa and easy sample processing.

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