Cumhuriyet Dental Journal

Volume 17 Issue 3

doi:10.7126/cdj.58140.5000016784

available at http://dergipark.ulakbim.gov.tr/cumudj/

RESEARCH ARTICLE

The effects of disinfectant solutions on the viscoelastic properties of acrylic resins

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ARTICLE INFO

Article history:
Received 09 May 2014
Accepted 25 June 2014

Keywords:
Acrylic resins
Disinfectant solutions
Dynamic mechanical thermal analysis
Viscoelastic properties

ABSTRACT

Objectives: The aim of this paper was to analysis the effects of disinfectant solutions on polyoxymethylene viscoelastic properties in comparison with polymethyl methacrylate.
Materials and Methods: Polymethyl methacrylate and polyoxymethylene acrylic and 200 mL 5% chlorhexidine gluconate, 2% sodium hypochlorite and one Corega Tabs dissolved in 200 mL water at 40°C were used. The storage modulus, loss modulus, Tanδ and glass transition temperatures of the disinfected acrylic resins were observed with the Thermal Dynamic Mechanical Analyzer.
Results: Lower storage modulus values were observed in the disinfectant solutions at polymethyl methacrylate specimens. The storage modulus showed lower values than the control group at chlorhexidine gluconate, higher values at sodium hypochlorite in the disinfectant solutions at polyoxymethylene specimens. While the glass transition temperatures of polymethyl methacrylate in the disinfectant solutions showed proximal values, the glass transition temperatures of polyoxymethylene in the chlorhexidine gluconate showed lower temperatures (approximately 11%).
Conclusions: The storage modulus, loss modulus, Tanδ values of polyoxymethylene were lower than those of Polymethyl methacrylate. The Corega Tabs did not affect the viscoelastic properties of polyoxymethylene. Chlorhexidine gluconate and sodium hypochlorite did not affect the viscoelastic properties of polymethyl methacrylate.

INTRODUCTION

Ethylene-derived thermoplastic acetal resins have recently come into use as alternatives to metal substructures for removable partial dentures. These resins are a type of polyoxymethylene (POM) that have a branchless, linear chain structure formed by the polymerization of formaldehyde originating from free acetal resin monomers.[1] Acetal resin is a polycrystalline structure and an injection-molded resin.[2] Acetal resin has been shown to have good physical and mechanical properties. The material has been also shown to have good biocompatibility that's why it was considered as a framework material for removable partial dentures for patients

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with allergic reactions to metal alloys. These properties make it an appropriate material for removable partial dentures, complete dentures, provisional bridges, occlusal splints, orthodontic and sleep apnea appliances.[3-6] Prolonged use requires that these materials exhibit chemical and mechanical resistance in their surrounding environments over time. Numerous factors affect the mechanical properties of resins, including polymer molecular weight; ratio of residual monomers, plasticizers and cross-linking agents; internal porosity of the polymer matrix; material thickness; finishing techniques; contact with chemical agents; and patient-related factors. Resin properties may also be affected by loss of soluble components or water sorption, tension and temperature changes related to hot and cold food intake during clinical use.[7-10]

The porous structure of acrylic resin as well as the presence of any surface scratches or pits allow microorganisms to easily attach themselves to an acrylic-resin denture. Thus, in addition to long-term contact with oral tissue, saliva, blood and water in the oral environment, an acrylic-resin denture also requires immersion in a cleaning solution for long periods of time. Disinfection is necessary in order to minimize cross-contamination between the patient, dental personnel and the denture.[11-13] McCabe et al.[14] stress the need for proper disinfection of all prostheses. This may be achieved using various chemicals, including glutaraldehyde, chlorhexidine gluconate, alkaline peroxides, alkaline hypochlorites, diluted acids and enzymes.[15] In addition, Gronitskey et al.[16] have suggested that sodium perborate products (Corega Tabs) may have an antimicrobial effect.

The disinfection process is expected to have no negative effect on denture materials; however, a number of studies have reported some disinfectant solutions to cause undesirable changes in the physical properties of the acrylic base.[17-19] Depending on the type and length of contact, disinfection agents may cause structural changes in the inner matrix of the polymer.[20,21] Chau et al.[22] reported that 1% sodium hypochlorite eliminated microorganisms from the denture, and another study found immersion in 1% sodium hypochlorite, 4% chlorhexidine gluconate, or 3.78% sodium perborate for 10 minutes produced no change in the transverse strength of heat-polymerized polymethyl methacrylate. Similarly, Angelillo et al.[23] stated that glutaraldehyde-based disinfectants provided effective disinfection without causing degradation in plastic and rubber materials**.** Ayaz et al.[24] al stated that the effervescent denture cleaners (sodium perborate, sodium bicarbonate) may be degradation in polymer structure of acrylic resins and change color of acrylic resin teeth.

In contrast, other studies have reported changes in the flexural strength of resin immersed in sodium hypochlorite and, with heat application, alkaline peroxide.[17,25] Shenet et al.[20] reported softening of resin surfaces exposed to glutaraldehyde alkaline disinfection with phenolic buffer for 2 hours, with the affect increasing with further exposure, and Peracini et al.[18] found that Corega Tabs caused a considerable reduction in the flexural strength of acrylic resin as well as a color change.

DMA is a method used to analyze the viscoelastic properties of polymeric materials through the application and measurement of controlled sinusoidal stress.[26-29] The null hypothesis was that different disinfectant solutions would affect the viscoelastic properties of POM and polymethyl methacrylate acrylic resin (PMMA). In order to better understand the effects of hygienic procedures on the viscoelastic properties of acrylic resin prosthetics, this study analyzed and compared the effects of 3 different disinfectant solutions on the viscoelastic properties of POM and PMMA acrylic resin using DMA.

MATERIALS AND METHODS

Two different types of acrylic resin [PMMA(QC-20 resin; De trey, Dentsplay, England) and POM (Dental D;Quatrotti, RovelloPoro, Italy)] and 3 different types of disinfectants [5% chlorhexidine gluconate (Dental D, Quatrotti, RovelloPoro, Italy); 2% sodium hypochlorite (Miyako do Brazil Id e Com; Guarrulhos, Brazil); CoregaTabs (Stafford Miller; Dungarvan Co, Waterford, Ireland)] were used in this study.

Acrylic specimens (20 mm × 10 mm × 4 mm) were prepared according to the manufacturers' instructions and divided into the following groups:

Aa (n=1) : PMMA/chlorhexidine gluconate
Ab (n=1) : PMMA/sodium hypochlorite
Ac (n=1) : PMMA/Corega Tabs
Ba (n=1) : POM/chlorhexidine gluconate
Bb (n=1) : POM/sodium hypochlorite
Bc (n=1) : POM/Corega Tabs
Ad (n=1) : PMMA/200 mL distilled water (control)
Bd (n=1) : POM/200 mL distilled water (control)

Disinfectants were dissolved in 200 mL distilled water at 40°C and thermocycled (MSCT-3 plus; Marcelo Nucci-ME, Sao Carlos, Brazil) at 5°-55°C with a 60-second dwell time. Each specimen was immersed for 10 minutes in one of the solutions, removed and rinsed for 2 minutes in distilled water. This procedure was repeated 30 times (to simulate approximately 6 months of denture usage).

A Thermal Dynamic Mechanical Analyzer (Perkin Elmer Pyris Diamond DMA; Model 983 MA) was used to measure the storage modulus (E'), loss modulus (E''), Tanδ and glass transition temperatures (Tg) of disinfected acrylic resin samples at N_2 atmosphere and temperatures ranging from –60 to +250°C with heating rate of 10°C/min. (POM specimens could not be observed above +160°C because of the melting point of the material).

DMA measurements are sensitive to micro-level structural changes and are capable of thoroughly analysing the viscoelastic behaviour of polymers with relatively few samples. Because of the small number of samples in the present study, a detailed statistical analysis was not performed. Similar recent studies of the viscoelastic properties of dental materials have also been conducted without performing statistical analysis.[30-34]

RESULTS

E' values are shown in Figure 1-3. Among PMMA specimens, E' values of specimens immersed in chlorhexidine gluconate (Aa) (Fig. 1) were similar to those of the control group (Ad), whereas E' values of specimens immersed in sodium hypochlorite (Ab) (Fig. 2) and in Corega Tabs (Ac) (Fig. 3) were lower than those of the control group. Among POM specimens, E' values of specimens immersed in chlorhexidine gluconate (Ba) (Fig. 1) were lower than those of the control group (Bd), whereas E' values of specimens immersed in sodium hypochlorite (Bb) (Fig. 2) were higher than those of the control group and E' values of specimens immersed in Corega Tabs (Bc)(Fig. 3) were similar to those of the control group.z

E'' values are shown in Figure 4-6. Instances of decreases in E' values appear as peaks in E'' values, with the first peak (β) in E'' values observed between +5° to +24°C and the second peak (α) between 120° to 125°C for PMMA, whereas POM peaks were observed between –10° to –20°C (β) and peak between 115° to 127°C (α).

Tanδ (E''/E') values are shown in Figure 7-9. PMMA Tanδ values peaked at approximately 120°C, whereas no marked peak was observed for POM. PMMA Tanδ values were similar for all disinfectant solutions and were lower than those of the control group. PMMA Tg temperatures were similar for all disinfectant solutions.

However, POM Tg temperatures were lower for chlorhexidine gluconate than for the other disinfectant solutions (Table I).

DISCUSSION

As a result of eating and drinking, acrylic resins are exposed to temperature change and periodic stresses on a daily basis. Depending upon its duration, stress can produce a temperature change in the resin, and as the temperature increases, the polymers may be transformed into a viscous structure. If the cross-linking occurring as a result of polymerization is incomplete, the release of side groups of polymer chains may be observed in the DMA spectrum.[27] The E' value determines its rigidity and depends upon its ability to store mechanical energy, while the E'' value is associated with the energy absorbed during dynamic deformation. Tanδ, that is, the

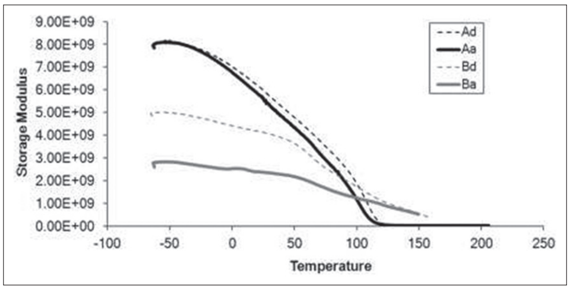

Figure 1. E' values of disinfectant solutions for chlorhexidine gluconate.

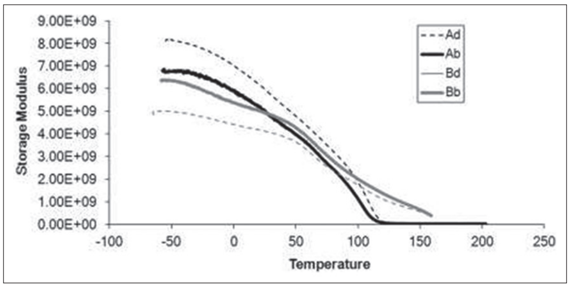

Figure 2. E' values of disinfectant solutions for sodium hypochlorite.

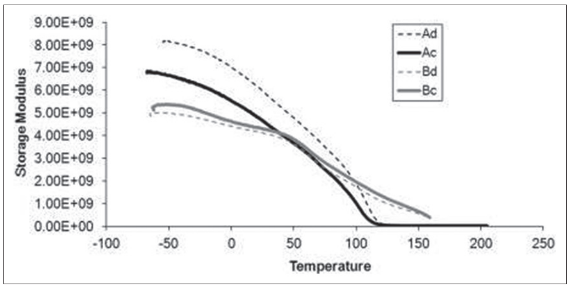

Figure 3. E' values of disinfectant solutions for Corega Tabs.

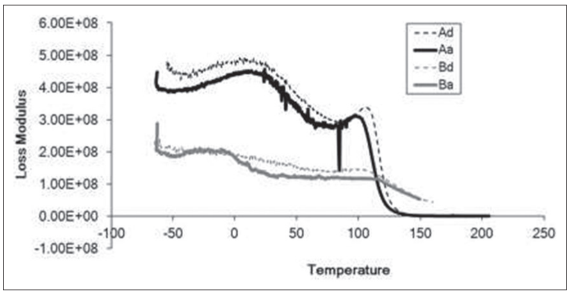

Figure 4. E'' values of disinfectant solutions for chlorhexidine gluconate.

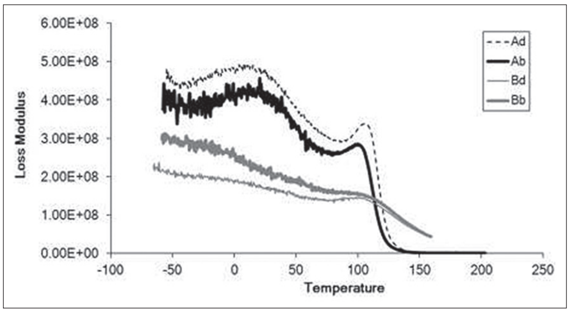

Figure 5. E'' values of disinfectant solutions for sodium hypochlorite.

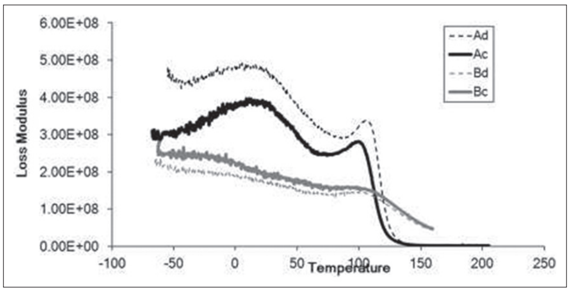

Figure 6. E'' values of disinfectant solutions for Corega Tabs.

Table 1. Glass transition temperatures (Tg) observed in the DMA spectrums.

Polymer groups	Aa	Ab	Ac	Ad	Ba	Bb	Bc	Bd
Tg (°C)	120.78	121.52	121.27	125.46	114.98	124.85	122.99	127.05

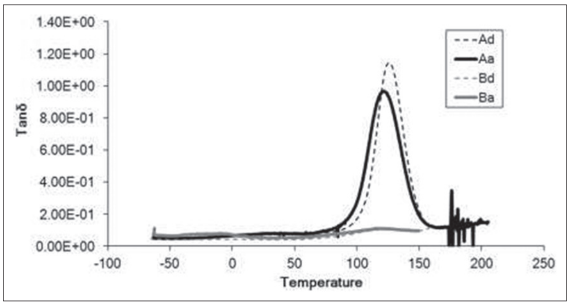

Figure 7. Tanδ values of disinfectant solutions for chlorhexidine gluconate.

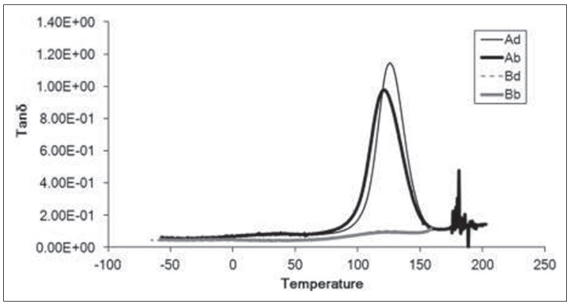

Figure 8. Tanδ values of disinfectant solutions for sodium hypochlorite.

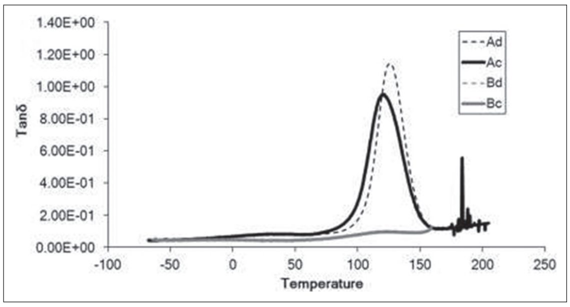

Figure 9. Tanδ values of disinfectant solutions for Corega Tabs.

ratio between E''/E', and the temperature at which polymer chains acquire the ability to move freely within a polymetric mass, represents the temperature at which the resin is transformed from a fragile glass with limited mobility to a totally fluid system.[32] Tanδ also provides an indication of the relative contribution of the elastic and inelastic components to the overall behaviour of the material.[29] A high tanδ indicates high molecular mobility, while a low tanδ indicates less mobility in the material. As temperature increases, the tanδ value and molecular mobility increases and the material approaches the rubbery.[35] Tanδ corresponds to Tg, a physical transformation in which a viscous or elastic material becomes a fragile glass. The midpoint of this transformation (the glass transition temperature) determines the temperature interval within which the material is suitable for use, with significant chain movements within the acrylic resin still occurring at the upper threshold of this interval. The Tg value of a polymer provides an indication of its cross-linking density.[26] and depends mainly upon the flexibility of the main chain. Other factors affecting Tg include the affinity between molecules, the addition of plasticizers or monomers with a different Tg, molecular weight, the amount of residual monomer and the density of polymerization. Moreover, any absorbed water acts like a plasticizer, affecting the affinities between the molecules, thereby lowering the Tg value and thus affecting the mechanical properties of the polymer.[33,34] Orsiet et al.[8] state that heating of the polymer resin that occurs during the polishing process creates a surface film that reduces water sorption, with residual monomers remaining within the internal porosities of the resin matrix. This residual monomer act as a plasticizer, adversely affecting the resin's mechanical properties and making it highly susceptible to deformation when subjected to stress.[7,9] Hamanaka et al[36] investigated the influence of water sorption on certain mechanical properties of injection-molded thermoplastic denture base resins. They reported that the water sorption significantly decreased the flexural strength and elastic modulus.

The main peaks in E'' and the tan δ curves show α relaxation in the main polymer chain. The peaks in the E'' curve at low temperatures indicate γ and β relaxation within the side groups and/or chain segments.[28] A relatively low tanδ value indicates low molecular mobility and high elasticity, whereas higher tanδ values indicates greater molecular mobility as well as viscosity.[37] In this study, the lower tanδ values (approximately 9 times in +70 to +160°C) observed for POM in comparison to PMMA. Thus, this study indicates POM to be an acrylic with greater elasticity.

PMMA specimens immersed in 5% chlorhexidine gluconate, 2% sodium hypochlorite and Corega Tabs exhibited similar Tg values, all of which were lower (approximately 3%) when compared to the control group. These similar Tg values indicate that crosslinking of the material inhibits the mobility of side groups, so that only the main chain exhibits movement in response to an increase in temperature. Moreover, the β transitions observed in the spectrums indicate a free release in molecules from the polymer chains, whereas α transitions are indications of cross linking in the material and mobility in the main chain but no release of molecules.[37] The use of 5% Chlorhexidine gluconate led to lower Tg (approximately 11%) in the POM. The use of 2% sodium hypochlorite and Corega Tabs led to lower Tg (approximately 3-4%) in the POM.

Cross-linked polymers have lower flexibility and higher Tg values than non-cross-linked polymer resins.[33] The differences found between PMMA and POM Tg values may be due to the fact that PMMA has a cross-linked structure, whereas POM is comprised of linear, branchless chains. According to Ruyter et al.[38] long distances between methacrylate groups contribute to more complete polymerization because of the high reactivity of the secondary methacrylate groups, which can be expected to contribute to a high degree of conversion and thus an increase in Tg. A decrease in the storage modulus value is an indication of an increase in flexibility.[39] While PMMA E' and E'' values showed no changes in 5% chlorhexidine gluconate, when PMMA was immersed in sodium hypochlorite and a Corega Tabs solution, both these values decreased, indicating a slight increase in resin flexibility. Previous studies have shown 1% sodium hypochlorite and 4% chlorhexidine gluconate to have no negative effects on the flexural strength or hardness of acrylic resin,[7,11] whereas CoregaTabs have been found to significantly reduce the flexural strength of acrylic resin.[18] Asad et al.,[17] however, found that immersion in 0.5% chlorhexidine gluconate for seven days did not result in any significant changes in the flexural strength of acryclic resins, but did lead to changes in surface hardness, whereas the flexural strength of non-cross-linked homopolymer resin specimens was significantly affected by immersion in alcohol-based disinfectants when compared to distilled water.

Peutzfeldt et al.[40] observed changes in acrylic surfaces as a result of contact with chlorhexidine digluconate and sodium hypochlorite, but not sodium perborate. The present study reported no changes in POM E' or E'' values with Corega Tabs, but found that POM E' values decreased with chlorhexidine gluconate, indicating an increase in flexibility, and that both E' and E'' values increased with sodium hypochlorite, indicating an increase in rigidity. Neppelenbroke et al.[15] stated that 4% chlorhexidine gluconate, 1% sodium hypochlorite and 3.78% sodium perborate all led to significant decreases in the hardness of acrylic resin; however, this effect was reversed following immersion in water for 15 days.

Pisani et al. investigated effect of cleaner solutions for denture cleaning on the properties of acrylic resin teeth. They found that 1% sodium hypochlorite caused alterations on the hardness, rougness and color of acrylic resin teeth.[41] Paranhos et al. reported that the alkaline peroxide and alkaline hypochlorite did not alter the flexural strength and 1% sodium hypochlorite solution caused increase in surface rougness of acrylic resin.[42] Campanha et al. evaluated the effect of long-term disinfection procedures on the hardness of acrylic resin denture teeth. 4% Chlorhexidine gluconate and 1% sodium hypochlorite and microwave sterilization decrease the hardness of the resin teeth.[43] Pisani et al. evaluated colour stability, hardness and rougness of acrylic soft denture liners after immersion in denture cleaners. They found that 1% sodium hypochlorite caused on increase in roughness.[44] Polyzois et al.[3] investigated the effect of peroxide and hypochlorite cleansers on gloss, colour and sorption of acetal denture resins. The immersion of acetal resin in NaOCl 5.25% showed clinically unacceptable and higher sorption and should be avoided or should be managed with care. Alaa Al-Haddad et al.[45] showned that Chlorhexidine reduced fracture toughness of the acrylic base (PMMA).

In this study, the E' values of PMMA specimens were higher (approximately 0.5 to 5 times) than those of POM specimens and PMMA specimen immersed in 5% chlorhexidine gluconate had the highest E' values (approximately 5 times). Thus, this result indicates that PMMA was more rigid acrylic than POM. The E'' values of PMMA specimens were higher (approximately 1.5 to 2.5 times) than those of POM specimens. The highest E'' values was observed in PMMA specimen immersed in 5% chlorhexidine gluconate.

The present study found slight differences in PMMA tanδ values observed among disinfectant solutions. These differences may be due to variations in the rate at which these solutions impact upon the resin surface film and thus differences in amounts of water absorption.

The null hypothesis was accepted because chlorhexidine gluconate and sodium hypochlorite affected the viscoelastic properties of POM, and Corega Tabs affected the viscoelastic properties of PMMA. The results of this study indicate that neither chlorhexidine gluconate nor sodium hypochlorite are suitable for cleaning POM prosthetics, whereas Corega Tabs should not be used to clean PMMA prosthetics.

The current study is limited in that only 2 type of acrylic resin and 3 type of disinfectant solutions are investigated. In order to identify the factors leading to changes in the Tanδ and Tg values of PMMA and POM, future studies should investigate factors such as polymer structure, cross-link density, water sorption and residual monomers.

CONCLUSIONS

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

1. E' (approximately 0.5 to 5 times), E'' (approximately 1.5 to 2.5 times) and tanδ (approximately 9 times) values of POM are lower than those of PMMA.
2. The use of Corega Tabs, 5% Chlorhexidine gluconate, 2% sodium hypochlorite disinfectant solutions led to lower Tg (approximately 3%) in the PMMA. The use of 5% Chlorhexidine gluconate led to lower Tg (approximately 11%) in the POM. The use of 2% sodium hypochlorite and Corega Tabs led to lower Tg (approximately 3-4%) in the

POM.
3. Corega Tabs do not affect the viscoelastic properties of POM and may be recommended for disinfecting prosthetics. 5% Chlorhexidine gluconate and 2% sodium hypochlorite do not affect the viscoelastic properties of PMMA; 5% Chlorhexidine gluconate and 2% sodium hypochlorite may therefore be usable as PMMA disinfectants.

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How to cite this article: Perihan Oyar, Gulsen Can. The Effects of disinfectant solutions on the viscoelastic properties of acrylic resins. Cumhuriyet Dent J 2014;17(3):205-214.