**Effect of food simulating agents on the flexural strength and surface hardness of denture base acrylic resins**

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**Abstract:**

**Aims:** The aim of this study was to investigate the effects of food simulating agents on the hardness and flexural strength of three acrylic resin materials.

**Methods:** Three acrylic resin materials (Meliodent, FuturaGen, hard GC reline) were used. Specimens for Flexural strength (FS) and hardness (VHN) determinations were fabricated in customized molds according to the manufacturers’ instructions. After polymerization, the materials were stored in the following FSA for 1 week at 370c: distilled water, 0.02 N heptanes, 75% ethanol solution, 0.02 N citric acid and lactic acid (n=10 per each group). For each test 10 specimens of each material were considered for baseline measurements (control). After conditioning, the FS and VHN were measured. Data were analyzed by one-Way ANOVA and Tukey Test (α=0.05).

**Results:** After water storage, only the VHN of FuturaGen specimens was decreased significantly, (p=0.000). Heptane conditioning decreased the FS of Meliodent and FuturaGen and also VHN of FuturaGen significantly (p<0.05). After lactic and citric conditioning, FS of Meliodent and FuturaGen and VHN of FuturaGen were significantly lower compared to the control (p<0.05). Hard GC reline was not affected by FSA except by ethanol solution. Among the FSA, 75% ethanol solution had the most adverse effect on the VHN and FS of the tested resin materials.

**Conclusions:** The mechanical properties of denture base acrylic resins could be influenced by food-simulating liquids. Therefore the clinician should caution patients regarding the possible effects of these agents on their denture especially if their prostheses are expected to function over an extended period of time.

**Keywords:** Denture base acrylic resins, Food-simulating agents Flexural strength, Hardness, Dental materials.

**1- INTRODUCTION**

Polymethyl methacrylate is one of the most commonly used materials in prosthetic dentistry and it has been used for denture base manufacturing since 1937. There are so many reasons to use heat-cured polymethyl methacrylate (PMMA) as a denture base material including its excellent esthetics, low water sorption and solubility, relative lack of toxicity, reparability and simple processing technique[1]. Compression molding technique is a standard method for curing resin. Simple processing, being familiar to dentists and technicians and also not needing any sophisticated or costly equipments make this method very useful. However there are some disadvantages such as dimensional changes and inaccuracies in the fit of the denture base. Therefore the popularity and relative simplicity of the compression molding technique are usually over shadowed by the high- processing stresses induced in the resin during polymerization [1,2].

Chemically activated or auto polymerizing resin is another type of resin which is not as frequently used for denture base fabrication as heat- activated resins. Nevertheless alternative materials such as FuturaGen, a cold cure PMMA, have been developed a breakthrough in denture base materials. FuturaGen has so many advantages including less shrinkage, less processing time, polishing simplicity, particularly smooth glass surface, exceptional denture adhesion characteristics, and significant plaque reduction as a result of its homogeneously smooth surface. In order to cure FuturaGen the injection molding technique is used which is similar to other injection methods. This technique is advantageous to the compression molding technique because it has less processing time and lower expenses and it causes less skin sensitivity to the evaporated monomer. Moreover the accessibility of the resin reservoir compensates for acrylic resin shrinkage [3].

Hard GC Reline is an improved methyl methacrylate-free acrylic resin which is used for chair side reline. This product produces less heat, odor and chemical irritation, high adaptability, less time-consuming complicated laboratory procedures, minimal porosity are some of the outstanding properties of this resin [4].

Mechanical properties such as flexural strength (FS) and hardness could be affected by the type of processing. FS is particularly important because acrylic resin removable dentures are susceptible to fracture after periods of clinical use. Some clinical factors for denture fractures include biting and mastication forces which have a deforming effect during function and any factor that increase the deformation of the base and changes the stress distribution. Midline fracture as a result of bending of complete dentures in the mouth is a frequently encountered problem [5].

On the other hand, it is claimed that hardness is sensitive to the residual monomer content in the polymerized resin [6]. One of the easiest ways to evaluate the degree of conversion of dental polymers is hardness [7], which is why hardness measurements have been successfully used as an indirect method to assess polymerization depth of resin- based materials [8] and the degree of conversion of conventional heat-polymerizing and self-curing acrylic resins [2]. Also hardness has also been used to predict the wear resistance of dental materials [9].

Dental restorations can degrade and age due to the existence of saliva; food components, beverages and interactions among these materials in the oral environment. Wu et al. [10] and Assmussen [11] reported that the resin matrix of dental composites soften when exposed to organic acids and various food and liquid constituents. In addition, when composites were soaked in oral fluids, disintegration at the resin filler interface occurred [12]. Several reports [13-16] have investigated the effects of food – simulating liquids on the mechanical properties of dental composites; however, to date, no studies have evaluated the effects of these agents on the mechanical properties of the resin denture base materials. Since, measurement of hardness and flexural strength (FS) together may be appropriate for evaluation of the clinical performance of these materials after exposure to food- simulating agents (FSA), the aim of this study was to investigate the effects of food- simulating liquids on the flexural strength and hardness of three different denture base acrylic resins.

**2- Materials and methods:**

Three types of denture base acrylic resins were selected for this study (Table 1). The tested acrylic resins materials were mixed according to manufacturers' instructions. For FS test a total of 150 specimens were fabricated using molds by investing brass dies of dimensions 65×10×3 mm according to ANSI/ADA specification No. 12.

The Meliodent and Futuragen specimens were processed by compression and injection molding technique respectively. In order to prepare hard GC reline the materials were injected into the molds. Subsequently, a glass slide was placed on top of the mold and a 10kg weight was applied to the glass to extrude excess material from the mold. Following the manufacturer's recommended polymerization time, the specimens were extracted from the molds. The specimens were inspected for the presence of air bubbles, and defective specimens were excluded from the study. Following the removal of flashes and trimming the edges, the specimens were ground with 320 grit size silicon carbide paper to obtain a polished surface. Prior to FS testing the dimension of each specimen was measured using digital calipers.

The specimen of each acrylic resin were randomly divided into five test groups and one control group, each consisting of 10 specimens. Specimens in the test group were conditioned for 7 days at 37 ˚C in the following storage solutions: distilled water, 0.02N citric acid, 0.02N lactic acid, 0.02 N heptane and 75% ethanol aqueous solution (Table 2). The control group was tested for baseline measurement. The food- simulating liquids used for conditioning the acrylic resins were chosen according to FDA guidelines [22]. Heptane simulates butter, fatty meats and vegetable oils. The aqueous ethanol solution and citric acid conditioning simulate beverages, including alcoholic drinks and vegetables, fruits, candy and syrup while water simulates the oral environment provided by saliva. The lactic acid simulates milk and dairy products. All the media were changed daily.

At the end of the conditioning period, the specimens were washed under running water, air- dried and the span and width of the specimen were measured using digital calipers. The specimens were subjected to a three-point bending test in a universal testing machine (Testometric, Testometric Co., UK) at a crosshead speed of 5mm/min and 50mm support span. The maximum load exerted on the specimens was recorded and FS values were calculated according to the following formula: S= 3PL/ 2WH2, where S is the flexural strength, P the maximum load applied to the specimen, L the span, W the width and H is the height of the specimen.

For hardness measurements, again a total of 150 specimens were produced in molds prepared by investment of brass dies (12×12×3 mm) within the flask. The materials were proportioned and processed according to manufacturer’s instructions. After polymerization, the specimens were inspected to have smooth surface without voids or porosity and were polished using progressively finer grade of silicon carbide papers.

The vickers hardness number (VHN) was determined for each specimen using a digital microhardness tester (Buehler MMT-3, Waukagen Lake Bluff, IL, USA). A 50gf load was applied through the indenter with a dwell time of 15 s. In order to accurately measure the indentation, a load of 10gf was applied for hard GC reline material, after the pilot study. The Vickers hardness number was measured at three different locations on each specimen and the mean was VHN value of each specimen. For statistical analysis, one-way ANOVA test and Tukey test were used to compare the variables between the groups, at a significance level of 0.05.

**3- Results**

Tables 3 and 4 show the mean FS and VHN of the denture base materials. In the control group FS and hardness of meliodent and futura gen specimens exhibited near values to each but Hard Gc reline specimens showed statistically significant lower FS and VHN than the other ones.

Fs of all specimens increased following immersion in water but the difference was not statistically significant in comparison with the control group (p>0.05). Hardness of Meliodent and Gc specimens in water increased insignificantly (p>0.05). For Futura gen the mean of VHN after storage in water was significantly lower than the control group (p = 0.000).

A statistically significant decrease occurred in the FS of Meliodent and Futura gen when heptane solution was used (p < 0.05), but no significant changes in Fs of Hard GC reline was observed in comparison with the control group (p=1.000). The heptane also significantly decreased the VHN of Futura gen (p = 0.000), but when compared to their control groups, no significant changes in VHN of Meliodent and Hard GC reline where noticed (p>0.05).

In the ethanol solution, the mean FS and VHN of all acrylic materials were significantly lower than their control groups (p < 0.05).

After conditioning in Citric acid and lactic acid, FS values of Meliodent and Futura gen were significantly lower than the control group (p < 0.05). Also acid storage caused significant decrease of Futura gen VHN in comparison with the base line measurements (p = 0.000) but Meliodent and Hard Gc did not show significant changes. After acid conditioning, insignificant increase in VHN value of Meliodent was observed (p=0.098).

**4- Discussion**

The current study was designed to determine the Vickers microhardness number (VHN) and flexural strength (FS) of three acrylic denture base materials following exposure to food-simulating liquids (FSL). The FSLs used for conditioning acrylic resins materials were chosen according to Food and Drug Administration guidelines (FDA, 1976, USA) [17]. Distilled water, Heptane, citric acid and ethanol solution, in addition to lactic acid were used to simulate the wet oral environment provided by saliva and water, butter and fatty meats and vegetable oils, certain beverages including alcohol, vegetables, fruits, candies, syrups in addition to milk and dairy products respectively. Within the oral cavity, acrylic resins may sporadically or constantly be exposed to the mentioned chemical agents. Sporadic exposure occurs in the course of eating or drinking until the teeth are cleaned. On the other hand, constant exposure occurs when the chemical agents are absorbed by adherent debris (for instance calculus or food particles) of restorations or produced by the bacterial disintegration of debris [16,18].

Since within the first 7 days of conditioning with these solutions, the most significant change in the hardness of resin composites was occurred [19], Prior to conducting the tests the specimens were conditioned in the FSL for 1 week. Although this period may seem long due to the fact that the restoratives only come into contact with foods and beverages for the duration of eating and drinking until teeth are cleaned, but these chemical agents can be stuck around the margins, under the denture, and into porosities of poorly manipulated materials. Moreover, calculus or food particles may also serve as reservoirs for these chemicals, leading to an increase in the exposure time of the restoratives to these agents [20]. Reports have shown that a regular drinker consumes 3.2 doses on a daily basis and each dose lasts for 15 minutes. The 24 hour storage time simulate one month of consistent drinking. As a result, 7 days immersion period represents 7 months consumption of that beverage [21].

Hardness is defined as the resistance of a material and its ability to abrade opposing dental structures. Strength, proportional limit and ductility are some of the properties associated with the hardness of materials. Hardness is a commonly used method to investigate factors that affect the degree of conversion of resins and to characterize mechanical qualities of a polymer due to the simplicity of specimen preparation and test method in addition to the availability of the equipment [7-9, 22].Therefore, in this study, we measured the hardness of one hard chair-side reline resin and two denture base resins to indirectly determine the degree of conversion. Following the storage of specimens in water and food simulating agents additional measurements were made in order to assess their effect on hardness properties. Moreover three point bending test was used to evaluate the flexural strength of specimens. This test evaluates a collection of properties such as tensile, compressive and shear strength and elastic modulus. Photo elastic analysis of stress shows that during mastication, denture is placed under Tensile and Compressive forces, therefore studying the flexural strength of denture base materials is important [23].

In the present study, hardness and Flexural strength value of Meliodent and Futura gen in the control group are almost close to each other and higher than GC which is similar to a study by Bahrani et al [24] The development of Futura gen, as cold cure PMMA, is considered a breakthrough in denture base materials. As stated by Futura gen manufacturer, the change in the initiator system in addition to using changed copper and barbituric acid ions, as a replacement for tertiary amine has lead to a reduction in the amount of residual monomer in this resin [24]. The low VHN and F.S value in GC in comparison to Futura gen and Meliodent may be due to the presence of high porosity, high levels of free monomer and lack of exposure to pressure during polymerization and existence of internal voids [25,26].

In agreement with some previous studies after immersion in water, FS of all specimens and hardness of Meliodent and Hard Gc increased insignificantly compared to their control groups [27-29] which could be related to the increase of the polymerization process [28], existence of free residual monomers and low water absorption. Residual monomer with its plasticizing effect [30-32], decreases the bonding of polymer interchain and causes deformation during the process of hardness testing [30,33].

Takahashi et al. [29] reported that water immersion had different effects on the flexural strength and hardness of different denture base and reline resin materials. They concluded that the results could be due to the fact that the intrinsic strength of the resin and the amount of water sorption in the system influences the mechanical strength of water absorbed acrylic resins. There are two processes that decrease residual monomer following polymerization: 1. Diffusion from the polymer, 2. More polymerization at the place of radicals in matrix [27,28]. At this point, acrylic is converted to a polar material that absorbs water by diffusion. Given that water molecules are smaller than the distance between the chains, the bond between the chains decreases, moreover it can act as a plasticizer, facilitating the flow of long chain polymers and reducing its chemical properties [34]. In addition the existence of cross-linking agents leads to a reduction in the amount of water sorption by resins during immersion. By filling micro voids, these molecules can exclude water uptake [35]. The presence of 1,6-hexanediol dimethacrylate in hard GC reline and ethylene glycoldimethacrylate in Meliodent as cross linking agents could cause low water sorption.

In a study by Azevedo et al. [1] 2 days of immersion in the water lead to a reduction in the hardness of the resin samples. As mentioned, water absorption and continuation of the acrylic polymerization process is time-dependent and diffusion-controlled. Studies have shown that both water [37] and residual monomer molecules [2] act as plasticizers, thus affecting the strength of polymerized resins. Hence we can conclude that the reduction in the hardness observed for resin materials during the first two days of water immersion is due to the more prominent plasticizing effect of water uptake than the released residual monomer molecules. But for immersion more than two days, like the current study, authors assume that these two factors could be the same and consequently hardness did not changed significantly.

In the current study, following the immersion in water, the hardness of Futura gen was decreased, in comparison with the control group, however, flexural strength was not changed, thus, it seems that the effect of water immersion on futura gen is limited to the surface rather than the bulk of the specimens. Neppelenbroek et al. [36] demonstrated that the type of disinfectant and the time of storage in water can affect the hardness of denture base resins [37]; however, the flexural strength of resins may not be affected by any of these solutions. They also revealed that the reduction in hardness related to disinfection procedures was reversed after storage in water for 15 days.

In this study, following conditioning of the resin samples in ethanol media, we noticed statistically significant decreases in the FS and VHN for all three acrylic resins in comparison with their control groups. PMMA dentures function in an acidic environment in alcohol drinkers. Assuming that a drinker consumes 1 to 2 h of alcoholic drinks on a daily basis, a total of 2000 to 3000 h effect the results for the 3-5 year life span of denture[38,39] . Vissidis et al.[38] stated that alcohol has two actions: first, it produces stress crazing at highly loaded positions of the dentures and thus reduces the static and dynamic strength of the base material and second, it causes corrosive effects on the surface of the denture. This may lead to an acceleration in fatigue processes within the denture material and subsequently cause premature failure. On the other hand Yap et al. [18] reported that partial removal of polymer matrix from its surface is the result of the destructive mechanism of ethanol, that is to say softening and damaging polymer matrix. The partial removal of the resin matrix may result in the degradation of the filler–matrix interface and also promote the release of reacted monomer. Although Vlissidis et al.[38] reported that over 4% alcohol in alcoholic drinks generate significant effects, it was shown that beverages with alcohol content as low as 25% could compromise longevity of resin-based materials [15] and this can contribute to the decrease in FS and KHN values [40,41]. As a result, it can be suggested that beverages which contain alcohol may compromise the functional longevity of the denture and the clinician should warn patients regarding the possible effects of alcohol on their denture particularly if their prostheses are expected to function over an extended period of time [42].

Results of a study by Yesilyurt et al[16] was in agreement with our study relating ethanol. Organic solutions may damage the resin matrix (heptane and aqueous ethanol solution).On the other hand, water and citric acids can damage organic fillers. Therefore organic solutions could decrease FS and hardness of dental resins.

In comparison with the control groups, only the FS of Meliodent and Futura Gen and hardness of Futura gen decreased significantly in heptane solution but no significant changes was observed in FS of Hard GC reline. This may be the result of the different chemical compositions of the materials; heptane could eliminate the leaching out of silica and combined metals in fillers, which occurs while conditioning in aqueous solutions such as dietary solvents [43]. Yap et al. [15] reported that after conditioning in heptane solution, Knoop Hardness Number (KHN) for methylmethacrylate based provisional materials decreased while KHN for bis-acryl resin-composite-based materials increased. Some studies on composites and provisional restorative materials found that heptane has no effect on the mechanical properties of composites with Bis-GMA. An earlier study also showed this phenomenon for several other commercial composite and compomer materials [13]. This finding can possibly be explained by the differences in the organic matrix composition between resin materials [16]. Conversely, heptane has a potential to damage some resin matrices [39]. In addition, the reduction of FS and VHN may also be attributed to the degradation of the inorganic filler [44]. It has been shown that the leakage of filler elements can generate cracks at the resin–filler interface [12], which may weaken the material. Furthermore, the contact surface of the resin matrix was identified as contributing factor. Solvent first exert a particular effect on the contact surfaces of resin matrices, before they proceed to penetrate the resin matrices fully [16].

Even though citric and lactic acids are weak intraoral acids, they reduced the FS of meliodent, futuragen and hardness of Futura gen, compared to the control groups. A possible explanation for this reduction could be the water sorption of polymeric materials following conditioning in this media. Other studies [45-47] on composite materials, claim that excessive water uptake can promote breakdown causing a filler–matrix debonding. The mechanical properties of materials may also be decreased by Silane hydrolysis and microcrack formation. Moreover, the reduction in FS can also be the result of the harmful effects of weak intraoral acids (citric and lactic acid) on inorganic fillers [39]. It has been shown that composites containing zinc and barium glasses are more susceptible to aqueous attack in comparison with quartz fillers [20].

In this study, the hardness and flexural strength of Hard Gc and hardness of Meliodent were not significantly changed after conditioning for 7 days in citric acid. However, a longer storage period may result in greater statistical significance. In addition, the harmful effects of acids are pH-dependent. Citric acid has a low acidic concentration of pH 2.6. Therefore, further studies are needed to investigate and elucidate the effects of citric acid conditioning on the hardness and flexural strength of acrylic resins. Hardness and FS of composite specimens in the Yesilyurt et al. study also did not significantly change after conditioning in citric acid [16].

Following immersion in citric acid an increase in the mean of VHN value was observed in Meliodent specimens in comparison with the control group. Other studies have also reported such an increase in composite and polyacid-modified composite and provisional restoratives [20,48]. However, to our knowledge there is no comparable study for acrylic resins materials. Further studies are needed to be conducted in order to have a more thorough understanding toward the increase in VHN after conditioning in acids.

In general change in mechanical and physical properties of dental materials after immersion in water and solvents may be due to the following factors:

1. **Chemistry of monomer resins:** For example an increase in water uptake occurred when the TEGDMA was used in resin matrix systems, since this monomer exhibits higher hydrophilicity when compared with Bis-GMA and UDMA [49].
2. **The extent of polymerization of the polymer matrix**: polymerization shrinkage and diffusion of moisture through the resin component lead to the initiation and propagation of micro cracks in the resin matrix. This process could provide a supply of chemical agents and a path for further diffusion into the restorative material, thereby resulting in more rapid degradation [44,50].
3. **Filler particle size, shape, and distribution**: For example it has been shown that composites which contain zinc and barium glass fillers are more susceptible to aqueous attack than those containing quartz fillers [43,48,51]. Yap et al.[18] stated that zirconia glass fillers were susceptible to aqueous attack as well.
4. **Interfacial properties between the filler and resin matrix [52,53]**.
5. **Contact surface of the resin matrix**: The contact surface of the resin matrix can be considered a contributing factor. Prior to fully penetrating the resin matrices, solvents apply a particular effect on the contact surfaces of resin matrices. Lack of oxygen inhibition layer on the surface subsequent to polymerization, leads to the low number of unreacted monomers on the surface. Incidentally, organic solvents promote the release of unreacted monomers and inorganic fillers in the resin matrix after penetrating the latter [42].

This invitro study could provide preliminary information the materials, based on bond strength and hardness test results. However further investigations are needed for longer periods along with clinical studies to assess whether other physical or chemical properties are influenced by the processing procedure or time involved.

5-**Conclusion:**

Within the limitations of this study, it may be concluded that the flexural strength and hardness of acrylic resins materials are influenced by food-simulating solutions especially aqueous ethanol solutions in vitro. These findings may provide support to clinicians to recommend restricted intake of certain beverages and foods to patients who have to use acrylic denture for an extended period of time.

**6-conflict of interest:** Not declared.

**7-Acknowledgment**

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