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EVALUATION OF SURFACE MICRO-HARDNESS AND FRACTURE TOUGHNESS OF CONVENTIONALLY CONSTRUCTED VERSUS CAD/CAM CONSTRUCTED DENTURE BASE MATERIALS- AN INVITRO STUDY

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ABSTRACT

The purpose of the current study was to evaluate surface micro-hardness and fracture toughness of Acetal Resin and Acrylic Resin (PMMA) constructed either by CAD/CAM milling method or by heat polymerized conventional method (lost wax method).

Materials and methods: Twenty-eight specimens (fourteen of each material); Acetal Resin and Acrylic Resin were constructed in the form of discs of 2mm thickness and 10mm diameter. Discs were divided equally according to the denture base material into two groups, group I for Acetal Resin and group II for Acrylic Resin (PMMA). Each group was subdivided according to the way of construction into; sub-groups (n=7). Sub-groups Ia and IIa for CAD/CAM milled discs and sub-groups Ib, IIb for conventionally constructed discs. Microhardness was measured on sample surface by digital display Vickers Micro-hardness Tester. Fracture toughness was evaluated by axial loading by the indentation technique.

Results: Group I (acetal resin group) recorded **statistically significant** higher microhardness mean values than group II (acrylic resin group). Moreover, group Ib (injection processed group) recorded **statistically significant** higher microhardness mean values than group IIb (*conventionally* processed group). Regarding fracture toughness there was **statistically insignificant** differences between all groups with higher mean values of group Ia,b (acetal resin groups).

Conclusion: CAD/CAM constructed acetal resin and acrylic resin denture base materials presented superior mechanical properties than conventionally constructed ones and expected to be more durable denture bases.

KEYWORDS: CAD/CAM, Acetal Resin. Acrylic Resin, microhardness, fracture toughness.

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INTRODUCTION

The production and the success of resins in dental field is always considered as a great step of dental materials development. Heat-cured acrylic resin materials was developed firstly in 1940s and has been used in the form of temporary crown and bridge restorations and as denture base material for removable prosthesis as partial and/or complete dentures. Acrylic resin is better known as polymethyl methacrylate (PMMA) and formed mainly of two components including polymethyl methacrylate powder and methyl-methacrylate monomer with a little percentage of crosslinking agent.¹ Thermal polymerization reaction of PMMA done by conventional lost wax technique method leads to unreacted monomers which cause toxicity, allergic reactions to the oral tissues and lack of resin matrix homogeneous structure, consequently, color changes of the material with weak mechanical properties could be noticed, moreover, thermally polymerized PMMA can results in highly porous material surfaces with increased water sorption properties accompanied with changes in material volume and difficulties in laboratory processing steps.²

Due to all this disadvantages, enhancement of polymer industry lead to production of new types of thermoplastic resins alternative to PMMA such as epoxy resins, polyamides (nylons), acetal resins (polyoxymethylene based resin materials), polycarbonate resins, polystyrene etc.^{3,4,5}

Since 1986, resins constructed via injection molds have been considered as a replacement of the conventional denture base and direct retainer materials mainly because of its superior esthetic properties.⁶ In the early 1990s, Acetal resin (polyoxymethylene based material POM) has been used to construct all partial denture framework components, denture bases, occlusal splints and implant abutments in addition to tooth colored clasps partial denture.⁷ Acetal resin offers superior esthetic properties in conjunction with more favorable physical and mechanical properties such as strength and flexibility with occlusal wear and fracture resistance, it is capable of restoring vertical dimension during provisional restorative treatment phase.^{8,9} However, on the other hand acetal resin lacks the natural color and translucency of thermoplastic resins and polycarbonate resins, it is technique sensitive material and requires special equipments.^{10, 11,12}

CAD/CAM (Computer-aided design and computer-aided manufacture) milling techniques have been introduced in dentistry for about four decades It can either involve additive manufacturing technique as rapid prototyping or subtractive manufacturing technique using computerized numerical control [CNC] machine. In prosthodontics, the subtractive procedure (CAD/CAM) is the more commonly used technique and it represents a recent way for designing, milling and constructing dental restorations, partial and/or complete dentures.¹³ There are various advantages of CAD/CAM fabricated dentures over the traditional dentures construction methods; for example: the denture can be designed and fabricated in less than three clinical appointments with accurate occlusal relationship that needs only minimal modifications or adjustments, moreover, the designs of the CAD/CAM constructed dentures can be digitally stored simply and effortlessly so the standardization of clinical researches on edentulism can be guaranteed.¹⁴

Superior physical and mechanical properties are considered as a dominant factor for the denture base materials to function successfully in the oral environment, denture base cracking or fracture is a commonly seen in the clinics either due to heavy occlusal forces and/or accidental base damage. Moreover, denture fracture may be also due to wrong denture design, improper fabrication method used.^{15,16}. Therefore, accurate and precise information are needed to understand the causes of denture base fracture and to improve its mechanical properties. So, the purpose of this study was to evaluate micro-hardness (VH) and fracture toughness of Acetal Resin and Acrylic Resin denture base materials constructed with CAD/CAM milling method and compare it with conventionally constructed methods.

MATERIALS AND METHODS

This study was done using two different materials divided into two groups; Group I used Acetal Resin denture base material which is considered as flexible resin and Group II Acrylic Resin denture base material which is a heat-polymerized type of resin. A total of 28 specimens; 14 specimens were constructed from each group and subdivided into 2 subgroups a and b (n=7) group Ia for CAD/ CAM milled Acetal resin specimens, group Ib for Acetal Resin specimens constructed by injection method, group IIa for CAD/CAM milled Acrylic Resin specimens and group IIb for Acrylic Resin specimens constructed by lost wax method.

Specimen fabrication:

A wax pattern in the form of disc of 2mm thickness and 10mm diameter was constructed using a silicon mold for standardization.

For groups Ia (CAD/CAM milled Acetal Resin)

The wax pattern discs were scanned using digital scanner (SHERA eco-scan 7, SHERA WORKSTOFF Technologies, Germany), then the standard tessellation language (STL) files contain the basic milling setting (specimens dimensions) were loaded onto the CAD software (FreeForm; Sensable) and then CAD/ CAM milled from Acetal resin (Bredent, Germany) and acrylic resin blanks (Ivoclar Vivadent Inc. USA) directly inserted in the same 5 axis milling machine (shera eco-mill 5 axis machine, SHERA WORKSTOFF Technologies, Germany). After completion of the milling, the blanks were removed from the machine and the

discs were retrieved finished and polished according to manufacturer instructions. Fig 1

Group Ib (injected method Acetal Resin discs)

Thermoplastic grains of Acetal Resin were heated inside its metallic cartridges then injected into a mold created by the specimen wax pattern, resin was injected by injection molding technique (Thermopress 400, Bredent GmbH & Co.KG, Germany) under very high pressure. The resin was plasticized under 220-265 °C and preheated for 15 min.¹⁷ Fig. 1

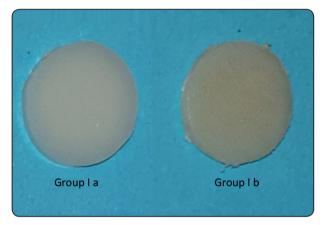


Fig. (1): Sample of acetal resin specimens (group I)

Group IIa (CAD/CAM milled Acrylic Resin)

Acrylic resin blanks (Ivoclar Vivadent Inc. USA) were used as in group Ia. Fig. 2

Group IIb (lost wax method acrylic resin discs)

Similarly, A wax pattern in the form of disc of the same dimensions (2mm thickness and 10mm diameter) was constructed from the previously mentioned silicon mold and flasked into the metal curing flask. After wax elimination procedure, the heat-cured acrylic resin (**acrostone, Egypt**) was mixed, packed and processed. When the polymerization cycle finished, the flasks were bench cooled, then all acrylic specimens were deflasked, finished and polished according to manufacturer instructions. Fig. 2

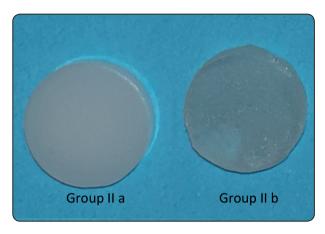


Fig. (2): Sample of acrylic resin specimens (group II)



Fig. (4): Indentations in group Ia

Testing Procedures

Surface Micro-hardness:

Surface Micro-hardness of all the specimens was measured by a device called digital display Vickers Micro-hardness Tester (Model HVS-50, Laizhou Huayin Testing Instrument Co., Ltd. China) with a Vickers diamond indenter and a 20X objective lens. Fig. 3

A 19.6 N load was used to the specimens surface for about 20 seconds long. Three indentations were made on each specimen surface, the indentations should be away from each other by about 0.5 mm or more and placed on equal distances. Fig.4,5,6,7



Fig. (5): Indentations in group Ib



Fig. (3): Vickers Micro-hardness Tester

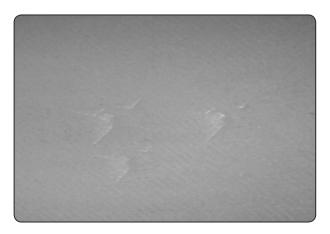


Fig. (6): Indentations in group IIa

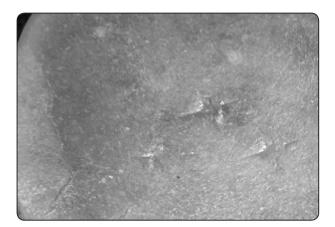


Fig. (7): Indentations in group IIb.

Built in scaled microscope was used to measure the indentations diagonal length, then Vickers values were turned into micro-hardness values through the following equation: **HV=1.854 P/d2**

where, **HV** is Vickers hardness in Kgf/mm2, **P** is the load in Kgf and **d** is the length of the diagonals in mm

Fracture toughness measurement:

The indentation technique used to determine and measure the fracture toughness depends on the formation of a series of cracks around the Vickers diamond indenter. The cracks appear to be emitted from the indentation corners. The length of these cracks "c" increased with an increasing the indentation load and is inversely proportional with the fracture toughness.

The fracture toughness was calculated with the following formula: KIC = 0.016(E/H)0.5(P/c1.5). Where KIC is the fracture toughness, **c** is the crack length (measured from the indentation center), **P** is the load, **H** is the Vickers hardness and **E** is the elastic modulus.¹⁸

RESULTS

The results were analyzed using Graph Pad Instat (Graph Pad, Inc.) software for windows. A value of $P \le 0.05$ was considered statistically significant with the satisfactory level of power set at 80% and

a 95% confidence level. Continuous variables were expressed as the mean and standard deviation. After homogeneity of variance and normal distribution of errors had been confirmed, two-way analysis of variance ANOVA was performed. One-way ANOVA was done for compared subgroups followed by student t-test.

Microhardness

Descriptive statistics of *microhardness* test results measured in (HV) showing mean values and standard deviation for all materials as function of processing technique are summarized in table (1) and graphically drawn in figure (8).

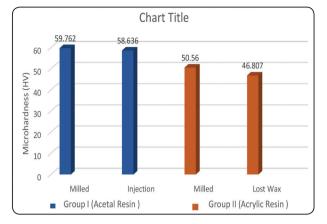
Regardless the processing method, it was found that group I (acetal resin group) recorded **statistically significant** higher microhardness mean values than group II (acrylic resin group). p=<0.0001<0.05 indicated by two-way ANOVA.

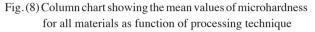
Irrespective of material group, CAD/CAM milled groups (Ia, IIa) showed **no statistically significant** differences with higher microhardness mean value of group Ia (acetal group), it was also found that, group Ib (injection processed group) recorded **statistically significant** higher mean values than group IIb (conventionally processed group) (p=<0.0001<0.05).

TABLE (1) Microhardness test results (Mean±SD)
	for all materials as function of processing
	technique

Variables		Statistics		t-test
		Mean	SD	P value
	Group Ia	59.762	1.1504	
Group I	Milled	39.762	1.1304	0.2852 ns
Acetal	Group Ib	58.636*	2.115	0.2032 118
	Injection	50.050	2.115	
	Group IIa	50,560	1.224	
Group II	Milled	50.500	1.224	0.0002*
Acrylic	Group IIb	46.807*	1.293	0.0002
	conventional	+0.007	1.275	
P value		0.0002*		

ns; non-significant (P>0.05) *; significant (P<0.05).





Fracture toughness

Descriptive statistics of *fracture toughness* test results measured in (MPa.m^{0.5}) showing mean values and standard deviation for all materials as function of processing technique are summarized in table (2) and graphically drawn in figure (9).

TABLE (2) Fracture toughness test results (Mean±SD) for all materials as function of processing technique

Variables		Statistics		t-test
		Mean	SD	P value
Group I	Group Ia			
Acetal	Milled	4.5377	0.56	0.1640 ns
	Group Ib			
	Injection	3.9277	0.6	
GroupII	Group IIa			
Acrylic	Milled	4.1836	0.95	0.1184 ns
	Group IIb			
	conventional	3.5249	0.5	
P value		0.1077 ns		

ns; non-significant (P>0.05) *; significant (P<0.05)

Regardless the processing method, it was found that group I (acetal resin group) recorded **statistically in-significant** higher fracture toughness mean values than group II (acrylic resin group) p = 0.1.07as indicated by two-way ANOVA.

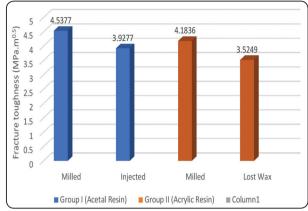


Fig. (9) Column chart showing the mean values of fracture toughness for all materials as function of processing technique.

Irrespective of material group, CAD/CAM milled groups (Ia, IIa) showed **no statistically significant** differences with higher fracture toughness mean value of group Ia (acetal resin group). It was also found that, group Ib (injection processed group) recorded **statistically in-significant** higher fracture toughness mean values than conventional processed group (p=0.1077 < 0.05).

DISCUSSION

In this laboratory study, methods of denture base construction and material composition and its role played on the final restoration mechanical and physical properties was evaluated by determining the microhardness and fracture toughness of acetal resin and acrylic resin denture base materials. The results showed significant higher surface hardness of Resin groups constructed by CAD/CAM milling technique than the resin groups constructed by conventional heat-polymerization technique, which might be due to the lower residual monomers content in CAD/CAM milled blocks. Many studies declared that acrylic resin denture bases which milled from blocks that have been polymerized under special condition of high pressure and elevated temperature values showed better surface properties compared to the conventionally constructed ones as the elevated pressure helps greatly in long polymer chains formation and consequently higher amount of monomer conversion with less residual monomer presence.^{19,20,21}

Another study confirmed that, different methods used for curing heat-polymerized resins seemed to have similar monomer conversion degrees, however, the use of higher pressure for longer periods of time bring a noticeable effect in increasing the monomer conversion into polymer.²²

In a study done to compare surface properties between conventional heat-polymerized and two new brands of pre-polymerized resin blocks used for CAD/CAM milling of complete denture, it was shown that, CAD/CAM Acrylic Resin demonstrated obviously better material surface properties including hardness and roughness, this may be related to the different way of construction of the CAD/CAM Acrylic Resin blocks which include elevated temperatures and pressure values used its polymerization than the conventional heatpolymerized ones.^{23,24}

Results of current study also showed that injection processed group recorded **statistically significant** higher mean values than *conventionally* processed group, this result goes with several other studies that revealed that injection molding techniques result in superiorly adapted and dimensionally accurate denture bases compared to the conventional techniques of fabrication.^{25,26}

The advantages of injection technique used in the present study were that the resin is delivered in a cartridge which reduces the errors results from improper powder/liquid ratio used, reduced of dimensional changes and helps in shape stability, as well as guarantee high physical and mechanical properties.²⁷

It was also found that injection method of processing showed higher microhardness and fracture toughness than conventional method of processing, this was also confirmed in 2012 with a study done by Farina et al, which reported that homogenous elevation of heating temprature of Acrylic Resin resulted in more monomer conversion into polymer, with less plasticizing effect of the residual monomers, and consequently superior surface hardness properties.²⁸

It was proved that residual monomers had hazardous effects and considered as a plasticizer which inferiorly affects the mechanical and the physical properties of the resin materials, also unreacted monomers trapped in and got surrounded by the formed polymer network and result in reduction of material clinical longevity and survivability.²⁹

It is well known that fracture toughness represents the resistance to cracking of the material. In this study, it was found that acetal resin groups recorded higher fracture toughness mean values compared to acrylic resin groups. This might be due to the high crystallinity nature of the acetal resin material structure (acetal resin (POM) is a semi-crystalline, thermoplastic polymer with high degrees of crystallinity)³⁰ which promote superior material mechanical and physical properties such as the hardness; the higher crystallinity structure present of the plastic, the harder and tougher it will be.³¹

this was proven by many other studies that the higher filler loading with wide distribution resulted in superior fracture toughness that can be reached a threshold value of ~55 to 57% filler loading. Moreover, fillers with higher volume showed superior surface hardness, compressive strength and also increased modules of elasticity. $^{32, 33, 34}$

In a study conducted to evaluate the value of adding ceramic fillers to PMMA denture base material and its effect on the material properties such as impact strength, the surface hardness and the fracture toughness of the resin, it was revealed that, the fracture surface of the PMMA without filler added showed of brittle fracture manner forming rapid cracks with sharp edges features, however, the manner ductile cracks formation were more obvious in the PMMA group with filles added, it could be concluded that, fracture resistance of heatpolymerized PMMA denture resin showed higher values after fillers addition.^{35,36}

CONCLUSION

Within the limitation of this study it can be concluded that;

- The method of construction of denture bases material had significant effect on its mechanical properties.
- 2- CAD/CAM constructed acetal resin and acrylic resin denture base materials presented superior mechanical properties than conventionally constructed ones.
- 3- Acetal resin groups showed higher microhardness and fracture toughness mean values than acrylic resin groups.

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