

## **The Influence of Different Chemical Surface Treatment on Transverse Strength of Repaired Heat Cure Acrylic Resins**

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**Abstract:** *Denture fracture is one of the most common problems encountered by the patients and prosthodontics. An attempt have been made to improve the mechanical properties of the repaired heat cure acrylic resin by chemical surface treatment. The aim of this study was to evaluate and compare the transverse strength of repaired conventional and hi-impact heat cure denture base resins with and without surface chemical treatment. .Four organic solvent which are available in the Iraqi market acetone, isopropanol, toluidine and ethyl acetate were used. A total of 100 specimens of  $64\pm 0.3 \times 10\pm 0.03 \times 2.5\pm 0.03$ mm dimensions were prepared, fifty specimens of each heat cure acrylic resin type (conventional and high impact), each fifty specimens were farther divided into five subgroups of 10 specimens as follow, control group (without surface treatment), surface treatment with organic solvent. The result of the study revealed that surface chemical treatment with acetone, ethyl acetate, isopropanol and toluidine improved the*

*transverse strength of repaired heat cure denture base when compared with control group. Tow-way ANOVA indicated that surface treatment had a significant influence on the transverse strength. It can be concluded surface chemical treatment with acetone, ethyl acetate, isopropanol and toluidine improved the transverse strength of repaired heat cure acrylic.*

## **Introduction**

Although polymethylmethacrylate has been widely used as a main component of denture base polymer for many years, this material is sometimes fractured or cracked in clinical use. One of the factors that causes fracture is considered to be low resistance to impact, flexural or fatigue [1]. There are many predisposing clinical factors for denture fractures [2-4]. These fractures are often related to poor fit of the dentures base, poorly balanced occlusion, problems in design and manufacturing of the denture [4], as well as inherent stress on the denture base that happens over time [5].

Denture fractures occur outside the mouth and inside the mouth. Impact failure outside the mouth and flexure fatigue failure in the mouth are two most important causes of fracture of denture base [6]. Outside the mouth, they often occur as a consequence of impact (accident) as a result of expelling the denture from the mouth while coughing, or simply of dropping the denture [2,7]. Inside the mouth the causes of denture fracture can be excessive bite force, improper occlusal plane, high frenal attachment, lack of balanced occlusion, poor fit or limitation in denture base material [8].

Denture fracture happens more frequently in midline of maxillary complete denture. Midline fracture as a result of bending of complete dentures in the mouth is a frequently encountered problem [2,9]. When in function midline fracture is the result of flexural fatigue failure caused by cyclic deformation of the base,

and is more likely to occur because flexure of the denture base occurs along the midline [10]. Denture repairs should have adequate strength, dimensional stability [11,12], good color match [12,13], are easily and quickly performed [7,12], are relatively inexpensive [12,13]. The repaired of fractured prosthesis can be accomplished using acrylic resin that are heat polymerized, autopolymerized, light polymerized, or microwave polymerized [2]. Although the conventional and microwave polymerized materials demonstrate superior strength, these materials require a significant amount of working time due to necessary packing and flasking procedures and also present the added risk of denture distortion by heat [14,15].

Autopolymerizing resin as a repair material seems to produce better strength than visible cure resin [16]. The use of autopolymerizing acrylic resin which allows for simple, quick repair, is the most popular. Autopolymerizing acrylic provides rapid and economic convenience to the patients. Also, fit of the denture repaired with self-curing resin was invariably much better than the fit of denture repaired with heat curing resins [17]. Attempts have been made to improve the mechanical properties of the repaired sites by changing either the joint surface contours [12, 18], the processing methods [12], optimizing the distance between repaired sites [18] reinforcing materials such as wires [19], glass fiber [20,21], or by using surface treatment [10,22]. Chemical or mechanical treatments change the morphology or surface chemistry of the acrylic resin base material to promote better adhesion [23]. Etchants such as acetone, methyl methacrylate, methylene chloride [23,24] are used for improving the bond strength between the base and the repair material, chloroform [25], ethylacetate [23,26], methylformate and methyl acetate [27,28], are used to improve the transverse strength of repaired autopolymerizing resin. In this study four organic solvents, available in Iraqi market have been used, which are acetone, ethyl acetate, isopropanol and toluidine, they were used as chemical surface treatment for two type of heat cure acrylic denture base (conventional and hi-impact) repaired with autopolymerizing acrylic resin.

The aim of this study was to evaluate and compare the transverse strength of repaired conventional and hi-impact heat cure denture base resins with and without surface chemical treatment with acetone, isopropanol, toluidine and ethyl acetate and to recommend combination having superior transverse strength in repair of denture base.

### Materials and Methods

All materials used in this study are listed in Table1.

#### Specimens grouping

A total of 100 specimens were prepared, fifty specimens of each heat cure acrylic resin type (conventional and high impact), each fifty specimens were farther divided into five subgroups of 10 specimens as follow, control group (without surface treatment), surface treatment with acetone, ethyl acetate, isopropanol and toluidine.

**Table 1: Materials used in this study**

Material	Trade name	Manufacturer	Lot number
Conventional heat polymerized acrylic	Triplex Hot	IvoclarVivadent Liechtenstein	N74750
Hi-impact heat polymerized acrylic	Vertex	TUV CERT Netherlands	X4133 P01(powder) X4111L06(Liquid)
Autopolymerized acrylic	Triplex Cold	IvoclarVivadent Liechtenstein	N69982
Dental stone	Elite model	Zhermack Italy	117344
Acetone	Acetone	LcBort India	33064
Ethyl acetate	Ethyl acetate	LcBort India	33064
Isopropanol	Isopropanol	LcBort India	33064
Toluidine	Toluidine	Fluka chemic,AG,CH Switzerland	9470

### Specimens preparation

Metal patterns with dimensions of  $64\pm 0.3 \times 10\pm 0.03 \times 2.5\pm 0.03$  mm (length, width, thickness respectively) were constructed for transverse strength test, according to the Specification No.12 of the American Dental Association [29] which was used as a guideline for preparing the specimens. The metal patterns were coated with separating medium (petroleum jelly) , invested in dental stone in the lower half of flask taking care that one half of the thickness was embedded in the stone put in base of the flask after setting of the stone separating medium(cold mold seal) was applied. The counter part of the flask was then assembled and another mix of dental stone was poured to complete flasking, Stone was allowed to harden for 60 minutes before the flask was opened.

The metal patterns were invested each time when the samples are to be prepared, the flask was then opened and metal patterns were removed from the mould carefully. After the application of separating medium, the resins were mixed according to the manufacturers' instructions, powder to liquid ratios were 21.0 g/10.0 ml, for Vertex; and 23.4 g/10.0 ml, for Triplex Hot. The monomer and polymer were mixed at once, allowed to reach a dough stage and placed into the moulds.

For Vertex, the flasks were immersed in water at 73°C for 90 min, rising the temperature to 100°C and maintaining the boiling for 30 min. Triplex specimens were polymerized by immersing the flasks in cold water, raising the temperature to 100°C and maintaining this temperature for 45 min (according to the manufacturers instruction for each type). Once the polymerization cycle was completed, the flask was allowed to slow cooling at room temperature followed by complete cooling of the flask with tap water before deflasking. The acrylic specimens were removed from the stone mould trimmed with acrylic bur, to get a smooth surface a stone bur was used followed by (120) grain size sand paper to remove any remaining small scratches with continuous water cooling.. The final measurements of the specimens were obtained

using the vernier. The specimens were conditioned for one week in distilled water at 37°C according to ADA specification No. 12 [29].

### Repair method

The intact acrylic specimens were invested in dental stone in metal flask (Figuer 1) and these formed the repair indices (Figuer2). The specimens were sectioned in the middle, 3 mm [5], were ground using metal disc . For all groups butt joint surfaces were produced, the surfaces of test specimens to be surface treated with chemical etchants was swabbed with chemical etchants for acetone for 30 sec [23] and ethyl acetate for 120 sec [22,30], while isopropanol, and toluidine, for a period of 5 seconds too, followed by rinsing with water and air drying, the specimens were returned and positioned into the same stone indices in such a way that a 3 mm gap [5] existed between the two sections of the specimen (Figuer3). Specimens were repaired with autopolymerizing acrylic resin using sprinkle on technique. The joint space was slightly overfilled to allow for polymerization shrinkage and finishing. All specimens were stored in water at room temperature for 48 hours before the test [29].

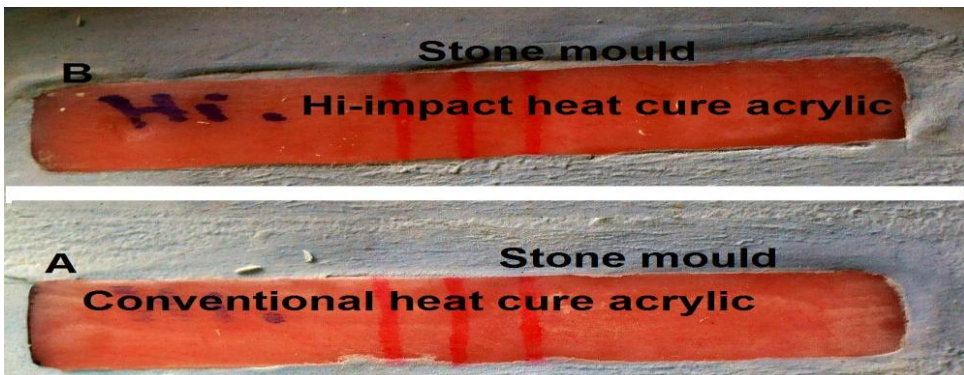


Figure 1: Acrylic specimens invested in dental stone.

A:conventional, B: Hi-impact

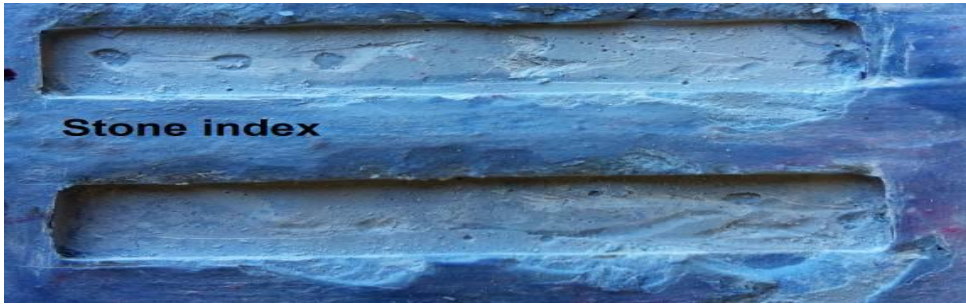


Figure 2: Stone repair index.

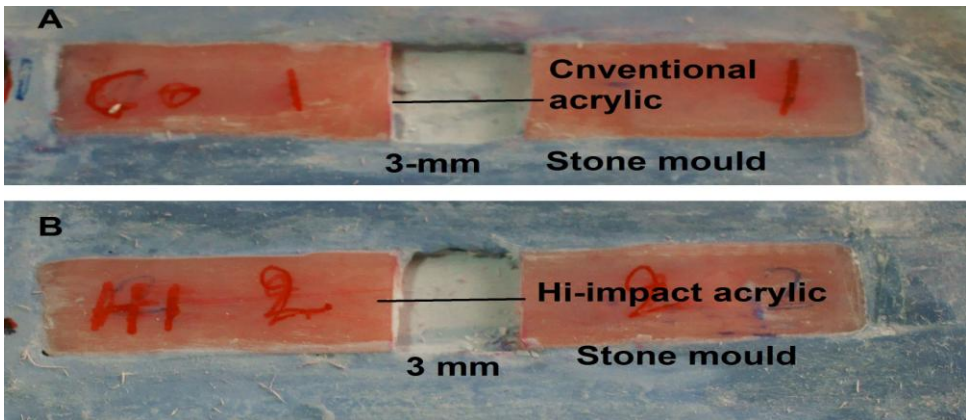


Figure 3: Sectioned specimens in stone indices.

## Testing

All specimens were tested for transverse strength with a three-point bending test with a universal testing machine (Hydraulic press, LeyboldHarris Co. British) using span length of 50 mm and crosshead speed of 5.0 mm/min. . The test consisted of gradually applying a force to each specimen until fracture occurred. The load was applied perpendicular to the center of the repaired area. The direction of the load was similar to the load direction that affects repaired maxillary denture. The maximum load applied was used to calculate the transverse strength by means of the specimens' cross sectional area, according the following equation [31]:

$$TS = 3Wl/2bd^2$$

Where:

TS= transverse strength (N/mm<sup>2</sup>).

W= load at fracture (N)

l= distance between supporting wedges (50 mm)

b= width of the specimen (10 mm)

d= thickness of the specimen (2.5 mm).

### **Statistical analysis:**

All statistical analysis were performed with the help of the statistical package for social scientists (SPSS) computer software for Windows versions 19.0. Mean values and standard deviations were calculated. Data were analyzed by two-way ANOVA and mean values were compared by using the Tukey (HSD) test. The level of statistical significance was established at  $p < 0.05$ .

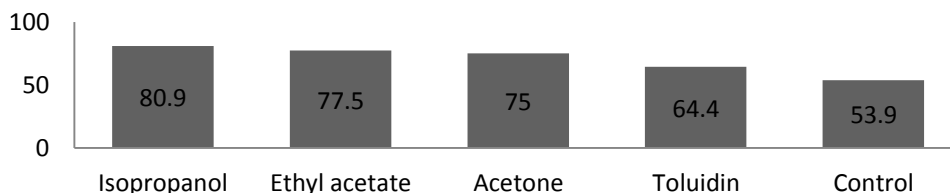
### **Results**

The mean transverse strength values and standard deviations for each type of acrylic base material and surface treatment are presented in Table 2. The study revealed that surface chemical treatment with acetone, ethyl acetate, isopropanol and toluidine improved the transverse strength of both conventional and hi-impact repaired heat cure denture base when compared with control group ( Figure 4) and transverse strength mean values of repaired hi- impact acrylic with and without surface treatment are higher than that of conventional acrylic resin (Figure 5).

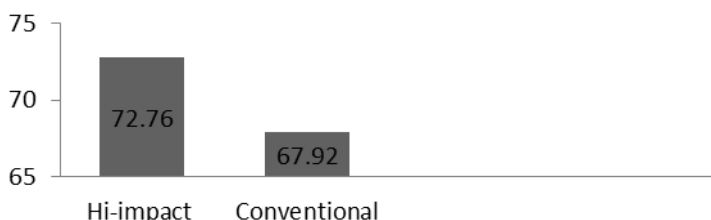


**Table2: - Mean Transverse strength values and standard deviation for acrylic resins and different surface treatment.**

Surface treatment	Acrylic material	Mean	Std. deviation	No.
Control (without surface treatment)	Conventional	48.000	8.344	10
	High impact	59.800	8.204	10
	Total	53.900	10.347	20
Acetone	Conventional	73.200	4.732	10
	High impact	76.800	4.821	10
	Total	75.000	5.631	20
Ethyl acetate	Conventional	74.600	5.330	10
	High impact	80.400	4.087	10
	Total	77.500	5.501	20
Isopropanol	Conventional	80.200	4.131	10
	High impact	81.600	5.059	10
	Total	80.900	4.552	20
Toluidin	Conventional	63.600	6.449	10
	High impact	65.200	4.732	10
	Total	64.400	5.566	20
Total	Conventional	67.920	12.252	50
	High impact	72.760	10.610	50
	Total	70.340	11.866	100



**Figure 4: Bar charts of transverse strength mean among surface treatment groups**



**Figure 5: Bar chart of transverse strength mean of repaired acrylic material.**

Tow-way ANOVA indicated that surface treatment had a significant influence on the transverse strength,  $F=69.182$ ,  $p<0.01$  and the type acrylic material has significant effect on the transverse strength as the mean value of transverse strength was significantly higher for repaired hi-impact acrylic denture base ( $M =72.760$   $SD =10.63$ ) than for repaired conventional acrylic denture base ( $M =67.92$ ,  $SD = 12.85$ ),  $F=16.535$ ,  $p < 0.01$ , On the other hand, significant differences were found in the behavior of the various base materials to the surface treatment,  $F= 92.585$ ,  $p < 0.05$  (Table 3).

**Table 3: Two-way ANOVA.**

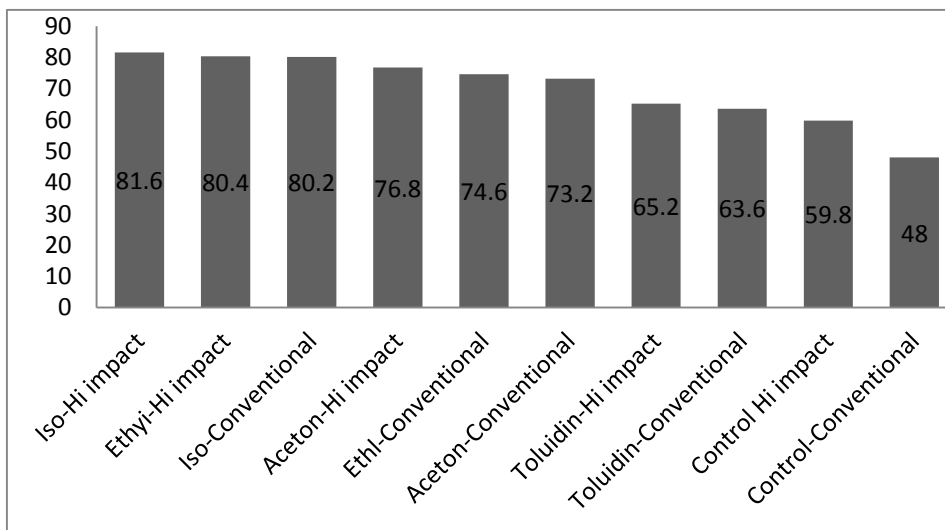
Source of variation	Sum of Squares	df	Mean Square	F	Sig.
Surface treatment	9801.040	4	2450.260	69.182	.000
Acrylic material	585.640	1	585.640	16.535	.000
Surface treatment * acrylic material	366.160	4	91.540	2.585	.042
Error	3187.600	90	35.418		
Total	508712.000	100			

Further analysis was carried out to see if any significant difference existed between any pairs of groups by the method of multiple comparisons Tukey test (Table 4). Thus, analysis

suggested isopropanol surface treatment has the highest transverse strength followed by ethyl acetate, acetone , toluidine and control group has the lowest mean transverse strength value which is significantly lower than that of surface treatment experimental groups  $p < .01$ .

**Table 4:** Multiple comparisons Tukey test for surface treatment groups.

Surface treatment	Surface treatment	Mean Difference	Std. Error	Sig.
Control	Acetone	-21.1000*	1.88196	.000
	Ethyl acetate	-23.6000*	1.88196	.000
	isopropanol	-27.0000*	1.88196	.000
	Toluidin	-10.5000*	1.88196	.000
Acetone	Ethyl acetate	-2.5000	1.88196	.674
	Isopropanol	-5.9000*	1.88196	.019
	Toluidin	10.6000*	1.88196	.000
Ethyl acetate	Isopropanol	-3.4000	1.88196	.376
	Toluidin	13.1000*	1.88196	.000
Isopropanol	Toluidin	16.5000	1.88196	.000



**Figure 6:** Bar charts of transverse strength mean among study groups

Hi-impact acrylic base material with isopropanol surface treatment is recommended as the combination processing the most superior transverse strength among the various combinations (Figure 6).

## Discussion

The material most commonly used for fabricating removable partial and complete dentures is heat-cured polymethylmethacrylate PMMA [19, 32].

Various reports have cited desirable properties of this polymer material, such as biocompatibility, adequate mechanical properties, satisfactory dimensional stability, insolubility in oral fluids, acceptable aesthetic, ease of handling and moderate cost. However, the strength of these bases remains far from ideal for maintaining the longevity of dentures [31, 33].

In order to develop stronger denture base materials resistant to fracture, different approaches have been proposed such as reinforcement of denture base materials with wire [34,35], woven polyethylene fiber [36], carbon fibers [37], glass fiber [38,39] and glass sheet or glass rod [40,41]. High-impact strength acrylics employ a PMMA polymer modified by adding a rubber compound to improve strength properties [42].

To compare transverse strength, two types of acrylic denture base were selected in the present study:

- 1- Conventional heat polymerization acrylic resin.
- 2- Hi- impact heat polymerization acrylic resin.

According to Craig [31] the repair materials are

1-Heat-accelerated acrylic resin:

- A) Water bath.
- B) Microwave

2-Chemically-accelerated acrylic resin

3-Light activated acrylic has been shown to be a fast and effective repair material

The material of choice will depend on the following factors:

- 1- Length of time required for making the repair.
- 2- Transverse strength obtainable with repair material.

- 3- Degree to which dimensional accuracy is maintained during repair.

Repairs performed with autopolymerized resin do not require much time and are inexpensive and easy to perform, autopolymerized resin was used as a repair material in this study due to above advantages.

The choice of denture base resin, as well as of the repair material may also influence the strength of the repaired denture [9,43,44]. Autopolymerizing acrylic resin has only 55% to 65% of the original heat cure denture strength<sup>26</sup>. Various methods for enhancing the strength of the repaired parts have been reported including repair surface designing, repair surface treatments and combined use of auto polymerizing acrylic resin with reinforcing materials such as metal wire and glass fiber [35,45].

Acetone, ethyl acetate, isopropyl and toluidin. are organic solvent. They were chosen as surface treatment chemicals in this study. Acetone is used as a solvent by the pharmaceutical industry and as a denaturant in denatured alcohol[46], Acetone is also present as an excipient in some pharmaceutical drugs., Acetone is used to synthesize methyl methacrylate [47].

Ethyl acetate is primarily used as a solvent and diluents, being favored because of its low cost, low toxicity, and agreeable odor. For example, it is commonly used to clean circuit boards and in some nail varnish removers [47]. Isopropyl alcohol dissolves a wide range of non-polar compounds. It also evaporates quickly and is relatively non-toxic, compared to alternative solvents. Thus it is used widely as a solvent and as a cleaning fluid, especially for dissolving oils [47]. Toluidin is a common solvent, able to dissolve paints, paint thinners, silicone sealants, many chemical reactants, rubber, printing ink, adhesives (glues), lacquers, leather tanners, and disinfectants [47]. No guideline were available from a previous study concerning the most effective application time of isopropanol and toluidine, five seconds of surface treatment was chosen as optimum time: as with increased time of treatment, the surface texture of acrylic becomes more porous which might compromise

the strength of repair. The application of isopropanol and toluidine to repair surfaces for different time periods need more investigations.

In regard to the distance between the repaired ends of the fractured parts, 3 mm gap was used. It was reported by Belyi and Vonfraunhofer [5], that the gap size between the broken pieces should be 3mm or less to minimize the bulk of repair material used, which will decrease the degree of polymerization contraction and reduce any color difference between the denture base and repair material [5]. One of the principal factors in the strength of repair is the type of joint used in repair. Beyli and Vonfraunhofer<sup>5</sup> noted that the butt joint repair was superior to the other joint type.. Butt joint type was used in this study. It is suggested to investigate the effect of acetone, ethyl acetate, isopropanol and toluidine on the transverse strength of repair denture base acrylic with rounded, and 45° bevel joint.

The transverse strength test, one of the mechanical strength tests, is especially useful in comparing denture base materials in which a stress of this type is applied to the denture during mastication [31]. It is a combination of compressive, tensile and shear strengths, all of which directly reflect the stiffness and resistance of a material to fracture [48,49]. The results of current study indicated that the transverse strength was influenced by surface treatment as transverse strength of surface treated repaired acrylic specimens was significantly higher than repaired acrylic specimens without surface treatment, this finding can be explained as follow:

Chemical surface treatment creates superficial crack propagation, as well as the formation of numerous pits approximately 2µm in diameter. This surface morphologic change may enhance the mechanical retention between a fractured surface and repaired acrylic resin. This may be attributed to superior adhesion because of monomer infiltration into the pits and cracks [50]. Mechanical strength of repaired denture base can be improved by pretreatment of surface with various chemicals such as chloroform, methylene

chloride, and ethyl acetate [10,20,51] . Shreya,et al reported that the increased transverse strength following methylene chloride and ethyl acetate surface treatment can be attributed to tight adhesion, which is the consequence of monomer infiltration into pits and cracks, surface treatment causes superficial crack propagation, as well as formation of numerous pits, the resulting surface morphology with pitting and elevation is caused by dissolution of polymethylmethacrylate by ethyl acetate and methylene chloride. This increase mechanical interlocking, farther improving adhesion between surfaces to be joined [26].

Findings of current study indicated that there was 38,8% increase in transverse strength after treatment with acetone in comparison to control group ( without surface treatment). Wetting the repair of acrylic specimens with acetone would wash away most of the micro debris and create a sponge like structure,these surface modifications improve the bond strength of the repaired material to the denture base [49] .These results are in accordance with ALnadawi LM ( 2005) [28] and Vojdani M, et al ,(2008) [16].According to the results of this study surface treatment with ethyl acetate provided 43.8% increase in transvers strength as compared to control group. George R and D'Souza M concluded that surface chemical treatment with ethyl acetate improves the repair strength of both heat cure and cold cure repair resins[51].. Shreya C et al <sup>26</sup> concluded that surface chemical treatment with ethyl acetate improved the transvers strength of repaired heat cure acrylic. Shimizu H et al [22] concluded that 120-second surface application of ethyl acetate enhanced the shear bond strength between the repair resin and the denture base resin, although the bond durability was inferior to that of the conventional surface preparation.

This study revealed that there was 50.2% increase in transverse strength after treatment with isopropanol as compared to control group which is the highest amount of increase among the four chemicals used in this study, this can be explained as isopropanol can form strong bonds (hydrogen bonds) with suitable

organic compound like methyl methacrylate, methyl groups in isopropanol act as electron releasing agent which increase the electron density on C2 in isopropanol so the electron- negativity on oxygen atom in C-OH in isopropanol will increase by inductive causing very strong bond with acrylic [52] (Figure 7).

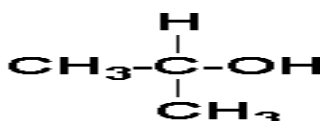


Figure 7: Isopropanol

present study revealed that surface treatment with toluidine provide 27% increase in transverse strength as compared to control group, .no previous study used this chemical as surface treatment, farther investigation is needed. Repaired hi-impact specimens (with and without surface treatment) had significantly higher transverse strength than repaired conventional acrylic specimens , this can be explained by the presence of rubber with acrylic as a co-polymer causing more hydrogen bonds and stronger bonds [52].

## Conclusions

With the limitations of this study, it can be concluded:

1. Surface chemical treatment with acetone, ethyl acetate, isopropanol and toluidine improved the transvers strength of repaired heat cure acrylic resins.
2. Transverse strength of repaired hi-impact acrylic significantly higher than that of conventional acrylic.
3. Hi-impact acrylic with isopropanol surface treatment have highest transvers strength among all study groups.

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## تأثير معالجة السطوح بالمواد الكيميائية على قوة الانثناء للقواعد الاكريليه المرممة

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### المستخلص:

انكسار الاطقم هي واحده من اهم المشاكل التي تواجه طبيب الاسنان والمريض. البحث الحالي اجري لتحسين الخواص الميكانيكيه للطقم المرمم بواسطة معالجات كيميائيه للسطوح المكسوره للاطقم. الهدف من هذا البحث هو تقييم ومقارنة المعالجات السطحية المختلفه على قوة الانثناء لمادتي الطقم , الاكريل العادي و الاكريل عالي المقاومة عندما ترمم بدون معالجة و بمعالجات سطحية كيميائية. أربعة مذيبات عضوية متوفرة في الاسواق العراقية استعملت في هذا البحث (أسيتون ، ايزوبروبانول ، توليودين و ايثايل اسيتيت). تم تحضيرمئة عينة بالابعاد  $64 \pm 0.3 \times 10 \pm 0.03 \times 2.5 \pm 0.03 \text{mm}$  خمسون عينة من كل نوع (الاکرل العادي و الاكرل عالي المقاومة) وكل مجموعة قسمت الى خمسة أقسام، عشرة عينات من كل نوع حسب نوع المعالجة الكيميائية. عشرة عينات من كل نوع أكريلك تعتبر مجموعة ضابطة بدون معالجة سطحية كيميائية. اظهرت النتائج ان المعالجات الكيميائية للأسطح بهذه المواد تزيد من قوة الانثناء عندما تقارن بالمجموعة الضابطة و لذلك يمكن أستنتاج بأن معالجة السطوح بالمواد الكيميائية المذكورة تزيد من قوة الأنثناءفي عملية تقوية الطقم المرممة.