Original Article

In vitro evaluation of transverse strength of repaired heat cured denture base resins with and without surface chemical treatment

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PURPOSE OF STUDY: Denture repair involves joining two parts of fractured denture with a denture repair material. The success of denture repair relies on the phenomenon of adhesion. Polymer surface can be etched by appropriate chemical, which changes the morphology and chemical properties of surface and promotes better adhesion. Taking into account the importance of adhesion in denture repair, the study was designed to evaluate and compare the transverse strength of repaired conventional, high-impact and glass fiber–reinforced heat cure denture base resins with and without surface chemical treatment with ethyl acetate and methylene chloride. **METHODOLOGY:** The study was conducted by surface treatment of different denture base resins (conventional, high impact, and glass fiber) with different chemicals (ethyl acetate and methylene chloride), with control group formed without surface chemical treatment. Specimens were repaired with autopolymerizing acrylic resin using 'sprinkle on' technique. The testing of the transverse strength of the repaired specimen was carried out on universal testing machine. **PRINCIPAL CONCLUSION:** The study revealed that surface chemical treatment with methylene chloride and ethyl acetate improved the transverse strength of repaired heat cure denture base when compared with control group. The glass fiber subgroup with methylene chloride surface treatment is recommended as the combination possessing the most superior transverse strength among the various combinations.

Key words: Repair, surface chemical treatment, transverse strength

INTRODUCTION

Impact failure outside the mouth and flexure fatigue failure in the mouth are two most important causes of fracture of denture base.[1]

Many different approaches to solving problems associated with broken dentures have been adopted in order to increase strength of the dentures after repair, such as modifying the denture material itself (high-impact resins) or reinforcing it with various fibers. Other methods involve various edge profiles, such as butt joint; 45 degree bevel rounded, knife edge, inverse knife edge, lap, rabbet, inverse rabbet and ogee joints; and joints with mechanical retention.[2,3]

The success of denture repair relies on the phenomenon of adhesion. Good bond should exist between the repair material and broken surface to be joined. Polymer surface can be etched by appropriate chemical, which changes the morphology and chemical properties of surface and promotes better adhesion.

Mechanical strength of repaired denture base can be improved by pretreatment of surface to be repaired with various chemicals such as chloroform, methylene chloride, and ethyl acetate.[4-6]

Aims and objectives

1. To evaluate and compare the transverse strength of repaired conventional, high-impact and glass fiber–reinforced heat cure denture base resins with and without surface chemical treatment with ethyl acetate and methylene chloride.
2. To assess mode of failure of fractured denture base.
3. To recommend combination having superior transverse strength in repair of denture base resins.

MATERIALS AND METHODS

- Acrylic resin specimen measuring 66 mm in length, 12 mm in breadth, and 2.5 mm in thickness was fabricated from wax specimen of the same measurement by process of acrylization.
- A custom-made rectangular mould 66 mm in length,

12 mm in breadth, and 2.5 mm in thickness was fabricated by investing the acrylic specimen into addition silicone elastomeric impression material of high viscosity [Figure 1].

- Molten wax was poured into mould and allowed to chill. Ninety wax samples were prepared in such manner. They were divided into 3 groups of 30 samples each.
- Group A consisted of wax specimens to be processed using conventional heat cure denture base resin.
- Group B consisted of wax specimens to be processed using glass fiber heat cure denture base resin.
- Group C consisted of wax specimens to be processed using high-impact heat cure denture base resin.
- The samples were then invested in plaster of Paris and dewaxed, after which they were left to cool at room temperature. Cold mold seal was then applied to the mould space. The mould space was packed with conventional glass fiber and high-impact denture base resin for group A, group B, and group C specimens respectively according to manufacturer's instructions.
- After curing and bench cooling to room temperature, the specimens were retrieved after deflasking. They were finished to a size of 64 mm length, 10 mm breadth, and 2.5 mm thickness according to American Dental Association specification no. 12 and stored in water.
- The prepared intact specimen was divided with the help of marker pen vertically into 2 equal parts, i.e., 32 mm each.
- One millimeter was marked on the right and left from center line on the top, and 3 mm was marked on the right and left from the center line at the bottom respectively.
- The prepared intact specimens were then vertically cut in accordance with the marking on the specimen.
- The surfaces to be repaired were ground with 800 grit silicon carbide sand paper.

Repair method

- **Stone index**: The intact acrylic resin specimens measuring 64×10×2.5 mm were invested in dental stone, and these formed the repair indices.
- Final groups were formed according to surface chemical treatment:
  Group 1: No surface pretreatment (control)
  Group 2: Methylene chloride surface pretreatment
  Group 3: Ethyl acetate surface pretreatment
- Ten specimens each of conventional denture base resin, glass fiber denture base resin, and high-impact denture base resin were divided in each group respectively.
  The following combinations in each group were thus obtained:
  Group 1: No surface pretreatment (control) on:
  1. Conventional acrylic denture base resin
  2. Glass fiber acrylic denture base resin
  3. High-impact acrylic denture base resin

  Group 2: Methylene chloride surface pretreatment on:
  1. Conventional acrylic denture base resin
  2. Glass fiber acrylic denture base resin
  3. High-impact acrylic denture base resin

  Group 3: Ethyl acetate surface pretreatment on:
  1. Conventional acrylic denture base resin
  2. Glass fiber acrylic denture base resin
  3. High-impact acrylic denture base resin
- After treatment of the cut surface, the heat polymerizing strips were fixed in mould to obtain a space for placing the resin to be repaired. Widths between strips were 2 mm at the top and 6 mm at the bottom [Figure 2].
- The surfaces of test specimens facing each other were swabbed with chemical etchant — methylene chloride in group 2, ethyl acetate in group 3, and no surface treatment in group 1 — for a period of 5 seconds, followed by rinsing with water and air drying.
- Specimens were repaired with autopolymerizing acrylic resin using 'sprinkle on' technique. The joint space was slightly overfilled to allow for polymerization shrinkage and finishing [Figure 3].
- All specimens were stored in water at room temperature for 48 hours before the test.

Testing

The testing of the transverse strength of the repaired specimen was carried out on universal testing machine [Figure 4]. The acrylic resin specimens were held in the fixture attached to the machine. Each specimen was subjected to the 3-point bending test at a crosshead speed of 5 mm/min at a 20-mm distance. 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 complete denture. The force required to fracture the denture base was recorded in kilograms.

The transverse strength (S) of each specimen was calculated using the following formula:

$$S = \frac{3WL}{2bd^2}$$

where
S = transverse strength,
W = load at fracture,
L = distance between end beams,
b = width of specimen, and

Figure 1: Prefabricated mould

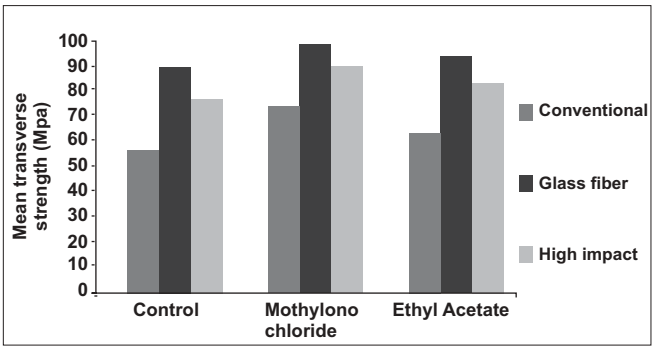

Figure 5: Multiple bar diagram showing mean transverse strength

Figure 2: Repair indices with specimen

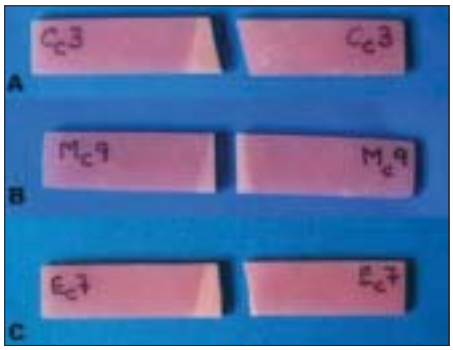

Figure 6: Fracture type — A: Adhesive fracture for control, B: Cohesive fracture for methylene chloride, C: Cohesive fracture for ethyl acetate

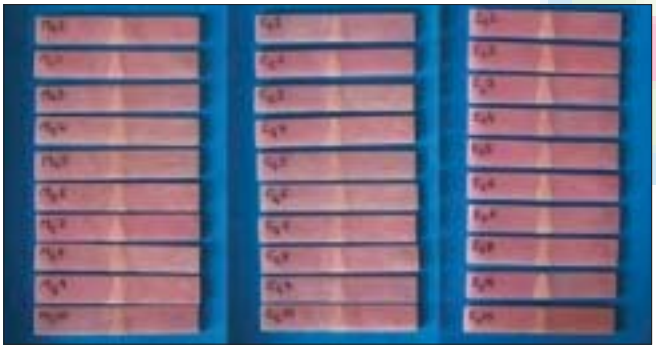

Figure 3: Repaired specimens

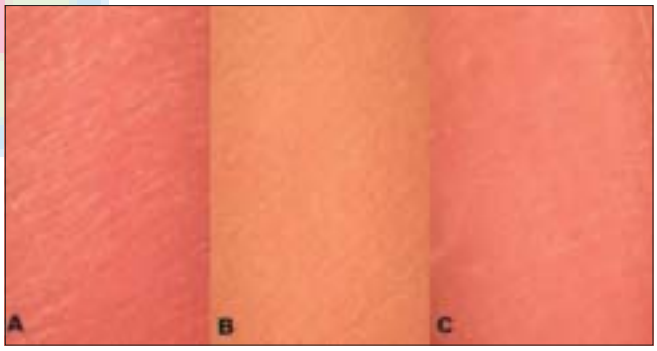

Figure 7: Adhesive fracture under light microscope for control group, A: Conventional heat cure denture base resin, B: High-impact heat cure denture base resin, C: Glass fiber–reinforced heat cure denture base resin

Figure 4: Transverse strength testing

d = thickness of the specimen.

The transverse strength in kg/mm^2 was converted to megapascals (Mpa) by multiplying it with 9.8 for converting it into the system of international units.

Force in kg/mm^2 × 9.8 = Force in Mpa

The fractured specimens were examined visually to determine whether the fracture was adhesive or cohesive.

Surface examination

The specimens which fractured at the interface were examined with a light microscope to determine whether the fracture was adhesive or cohesive.

A layer of pink autopolymerizing resin on repair surfaces of a test specimen of heat cure acrylic resin

was inferred as cohesive failure.

RESULTS

All statistical analyses were done with the help of the statistical package for social scientists (SPSS Inc., USA) computer software for Windows versions 10.5.

Values of transverse strength for control, methylene chloride, and ethyl acetate surface treatment are presented in Tables 1 to 3 respectively. The mean strength with standard deviation for 3 comparative groups (control, methylene chloride, and ethyl acetate) is presented in Table 4. The study revealed that surface chemical treatment with methylene chloride and ethyl acetate improved the transverse strength of repaired heat cure denture base when compared with control group. Two-way ANOVA revealed that there was statistically significant difference in mean strengths of the three groups and subgroups [Table 5]. Further analysis was carried out to see if any significant difference existed between any pairs of groups and subgroups by the method of multiple comparisons Bonferroni test [Table 6]. Thus, analysis suggested methylene chloride surface treatment has the highest transverse strength, followed by ethyl acetate surface treatment, and control group has low mean transverse strength [Figure 5].

The glass fiber subgroup with methylene chloride surface treatment is recommended as the combination possessing the most superior transverse strength among the various combinations.

The mode of failure in methylene chloride and ethyl acetate group is observed to be cohesive, suggesting tight adhesion of the autopolymerizing and heat polymerizing

Table 1: Control (no surface chemical treatment)

Conventional heat cure denture base resin	Glass fiber reinforced heat cure denture base resin	High impact heat cure denture base resin
52.8	81.41	76.8
55.87	92.16	76.8
51.26	84.48	79.87
58.94	89.09	69.12
58.94	84.48	69.12
52.8	92.16	76.8
51.26	86.02	72.19
58.94	84.48	70.66
51.26	92.16	76.8
52.8	90.62	69.12

Table 2: Methylene chloride surface chemical treatment

Conventional heat cure denture base resin	Glass fiber reinforced heat cure denture base resin	High impact heat cure denture base resin
67.58	90.62	84.48
64.51	92.16	92.16
67.58	99.84	89.08
69.12	96.77	87.55
76.8	92.16	86.11
69.12	95.23	89.08
79.87	99.84	86.11
70.66	99.84	86.11
76.8	96.77	86.11
69.12	99.84	92.16

Table 3: Ethyl acetate surface chemical treatment

Conventional heat cure denture base resin	Glass fiber reinforced heat cure denture base resin	High impact heat cure denture base resin
61.44	92.16	84.88
61.44	92.16	81.41
53.76	89.09	76.8
64.51	89.09	76.8
69.12	90.62	79.87
61.44	86.02	76.8
61.44	92.16	79.87
64.51	95.23	79.87
61.44	95.23	84.48
53.76	92.16	82.94

Table 4: The mean strength with standard deviation for 3 comparative groups (control, methylene chloride, and ethyl acetate)

Group	Subgroup	Mean	Std. deviation	N
Control	Conventional	54.4856	3.35350	10
	Glass fiber	87.7056	3.99557	10
	High impact	73.7280	4.09600	10
	Total	71.9731	14.33579	30
Methylene chloride	Conventional	71.1166	4.96627	10
	Glass fiber	96.3068	3.62470	10
	High impact	87.8950	2.66072	10
	Total	85.1061	11.28523	30
Ethyl acetate	Conventional	61.2864	4.66173	10
	Glass fiber	91.3920	2.82761	10
	High impact	80.3726	3.05352	10
	Total	77.6837	13.11968	30

Table 5: Two-way ANOVA

Source of variation	Sum of squares	df	Mean square	F ratio	*P* value
Group	2601.814	2	1300.907	83.45	.001
Subgroup	13319.97	2	6659.987	427.26	.001
Error	1324.94	85	15.58753	M	
Total	17246.73	89			

Table 6: Multiple comparisons Bonferroni test for groups

Group	Group	Mean difference	Std. error	*P* value.
Control	Methylene chloride	-13.1331	.97343	.0001
	Ethyl acetate	-5.7106	.97343	.0001
Methylene chloride	Control	13.1331	.97343	.0001
	Ethyl acetate	7.4225	.97343	.0001
Ethyl acetate	Control	5.7106	.97343	.0001
	Methylene chloride	-7.4225	.97343	.0001

acrylic resins; whereas in the control group, adhesive type of fracture was noted [Figure 6].

DISCUSSION

Acrylic resins have been extensively used for the fabrication of denture bases because they provide a large number of advantages compared to other materials. But one of the major drawbacks to the use of acrylic as denture base material is its susceptibility to fracture, which causes inconvenience and embarrassment to the dentist and the patient.

To compare transverse strength, three types of denture bases were selected.

1. Conventional denture base
2. Glass fiber reinforced denture base
3. High impact denture base

Osberone used strengtheners such as wires, nylon, and glass fibers. He found that the most efficient strengthener was glass fiber.[7] Also, glass fiber has improved transverse strength compared to aramid and nylon.[8] Problems associated with color and reduced strength have been largely overcome with introduction of glass fibers.

Denture repair involves joining two parts of fractured denture with denture repair material. In repair, focus is mainly on:

1. Type of repair material
2. Preparation of fractured edges
3. Repair surface treatment

Type of repair material

Broken acrylic dentures are repaired with:

a) Autopolymerizing acrylic resin
b) Heat cure acrylic resin
c) More recently, visible light cure resin

The visible light cure materials exhibit lower repair strength and toughness than does autopolymerizing resin.[9]

A denture repaired with heat cure resin exhibits approximately 85% of its original strength; however, dimensional changes are more in heat cure resin compared to autopolymerizing acrylic resin. The heat cure dentures exhibited considerable changes in contour after they had been repaired by the heat-curing method, but relatively no changes resulted

from the self-curing repairs.[10]

The use of autopolymerizing acrylic resin, which allows for simple, quick repair, is most popular. Autopolymerizing acrylic resin provides rapid and economic convenience to the patients. Also, fit of the denture repaired with self-curing resins was invariably much better than the fit of denture repaired with heat-curing resins.[11] However, autopolymerizing acrylic resin has only 55% to 65% of the original heat cure denture strength. Autopolymerizing acrylic resin was used as a repair material in this study due to added advantages.

Preparation of fractured edges

One of the factors in the strength of repair is the type of joint used in the repair. Various authors have indicated smooth and rough interface surfaces; butt joints; 45-degree angle joints; tapered and rounded joints; and joints with mechanical retention.

Harrison WM, Stansbury BE studied the effect of joint surface contours on the transverse strength of repaired acrylic resin. Three types of joint contours, viz., round joint, rabbet joint, and butt joint, were studied. They came to the conclusion that the rounded joint is superior to the rabbet and butt joints since stresses are uniformly distributed by preparing a rounded interface surface.[3]

The values of transverse strength of repairs made with round and 45-degree bevel joint designs are similar and significantly greater than those made with a butt joint design.[2]

Beyli studied the transverse strength of repaired acrylic resin. He used knife-edged, inverse knife edge, round lap, rabbet, and inverse rabbet joint surface preparations. The traditional butt joint for repair of fractured dentures has been found to be inferior to the inverse knife edge, round lap, and inverse rabbet joints. No significant differences were found between these five profiles.[3]

Joint configuration used in the study was a modification of butt joint such that joint space was 2 mm on top and 6 mm at the bottom. This design was used to study if length affects the type of fracture (adhesive or cohesive) or it's the surface chemical treatment which determines the type of fracture.

Repair surface treatment

Attempts to improve strength by chemical or mechanical modification of denture base resin have been described.

Mechanical modification includes grinding with burs, retention grooves, airborne particle abrasion and lasing to increase surface area, and mechanical retention to enhance van der Walls force of attraction. Because monomer is not a powerful solvent for polymethylmethacrylate, painting or immersing the surface will not efficiently remove the debris and create particle-free surface for bonding. Hence treatment with chemicals is essential.[4-6]

Mechanical strength of repaired denture base can be improved by pretreatment of surface with various chemicals such as chloroform, methylene chloride, and ethyl acetate.[4-6]

Shen C *et al.* concluded that treating the fractured denture surface with chloroform improves the quality of bonding. It is demonstrated by the various degrees of improvement in repair strength. Statistically significant improvement in strength is observed only when heat-cured resin is used.[4]

However, chloroform is found to be carcinogenic.

Reinforcement with glass fiber and methylene chloride pretreatment produced transverse strength and modulus of elasticity higher than those of control.[5]

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, with marked improvement for the heat cure resins.[6]

Toxicity of methylene chloride and ethyl acetate is low, which augments its role in surface chemical treatment for denture repair.

The study revealed that surface chemical treatment with methylene chloride and ethyl acetate improved the transverse strength of repaired heat cure denture base when compared with the control group. 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 increases mechanical interlocking, further improving adhesion between surfaces to be joined.[4-6]

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.

Type of fracture

For all the specimens, the interface where failure occurred was classified as either cohesive or adhesive in nature. The type of failure was identified to be adhesive when it fractured at the interface and cohesive when layer of repair resin was present on test specimen of heat cure acrylic resin.

In the control group, all samples fractured at the interface, i.e., adhesive type of fracture was observed.

In methylene chloride and ethyl acetate group,

cohesive type of fracture was noted.

In control group, samples which fractured at the interface were observed under light microscope to confirm if fracture was adhesive or cohesive in nature. No layer of pink autopolymerizing resin on repair surfaces of a test specimen of heat cure acrylic resin was inferred as adhesive failure. Light microscope confirmed that the fractures were adhesive in nature [Figure 7].

CONCLUSION

The study revealed that surface chemical treatment with methylene chloride and ethyl acetate improved the transverse strength of repaired heat cure denture base when compared with the control group.

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Source of Support: Nil, **Conflict of Interest:** None declared.