

## Argon Ion Laser Polymerized Acrylic Resin: A Comparative Analysis of Mechanical Properties of Laser Cured, Light Cured and Heat Cured Denture Base Resins

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### Abstract:

**Background:** Dentistry in general and prosthodontics in particular is evolving at greater pace, but the denture base resins poly methyl methacrylate. There has been vast development in modifying chemically and the polymerization techniques for better manipulation and enhancement of mechanical properties. One such invention was introduction of visible light cure (VLC) denture base resin. Argon ion lasers have been used extensively in dentistry, studies has shown that it can polymerize restorative composite resins. Since composite resin and VLC resin share the same photo initiator, Argon laser is tested as activator for polymerizing VLC resin. In the Phase 1 study, the VLC resin was evaluated for exposure time for optimum polymerization using argon ion laser and in Phase 2; flexural strength, impact strength, surface hardness and surface characteristics of laser cured resin was compared with light cure and conventional heat cure resin.

**Materials and Methods:** Phase 1; In compliance with American Dental Association (ADA) specification no. 12, 80 samples were prepared with 10 each for different curing time using argon laser and evaluated for flexural strength on three point bend test. Results were compared to established performance requirement specified. Phase 2, 10 specimen for each of the mechanical properties (30 specimen) were polymerized using laser, visible light and heat and compared. Surface and fractured surface of laser, light and heat cured resins were examined under scanning electron microscope (SEM).

**Results:** In Phase 1, the specimen cured for 7, 8, 9 and 10 min fulfilled ADA requirement. 8 min was taken as suitable curing time for laser curing. Phase 2 the values of mechanical properties were computed and subjected to statistical analysis using one-way ANOVA and Tukey *post-hoc* test. The means of three independent groups showed significant differences between any two groups ( $P < 0.001$ ).

**Conclusion:** Triad VLC resin can be polymerized by argon ion laser with 1 W/mm<sup>2</sup> power and exposure time of 8 min to satisfy ADA specification. Impact strength, surface hardness of laser cure

was better than light cure and heat cure resin. Flexural strength of light cure was better than laser cure and heat cure resin. The SEM study showed similar density on surface, the fractured surface of heat cure resin was dense and compact.

**Key Words:** Argon ion laser, denture base resin, flexural strength, hardness, impact strength, laser, light polymerization, scanning electron microscope study, visible light cure resin

### Introduction

Dentistry as a branch of science, has witnessed a continuous search for new materials and techniques, ever since its evolution, to improve the quality of treatment service. The practice of dentistry, in particular prosthetic dentistry is at an interface delicately balancing technology and science on one side and patient oriented service on the other. Technological innovations are heralding in changes at an ever increasing pace and pose a challenge to the clinicians.

Prosthetic dentistry in the last few decades has seen impressive developments with the introduction of new materials and perfection of already existing materials with astonishing results. Poly methyl methacrylate resin which was introduced in the year 1937 is being used extensively in the field of prosthetic dentistry.<sup>1</sup> No other material enjoys such a vast usage and acceptance in dentistry for its physical and esthetic properties and also for ease of manipulation, economical and material availability so much so that having an alternate material appears to be not possible in the near future and the only improvement would be the betterment of the existing material and technique for better use in the patient service.<sup>2,3</sup> One such attempt at improvement was the introduction of light curable denture base resin in 1983.<sup>4</sup>

This class of resin, which is polymerized by visible light consists of urethane di-methacrylate matrix with acrylic copolymer as filler, micro fine silica and photo initiator system instead of methyl methacrylate monomer, having advantage of zero residual monomer and also eliminates lengthy lost wax technique of investing, flasking and boil out required for conventional denture construction.<sup>5</sup> This material advocated for wider application in removable, fixed and maxillo-facial prosthodontics and orthodontics<sup>4,6</sup> because of its biocompatibility, ease of fabrication, color stability, improved physical properties and low bacterial adherence due to its low surface porosity.<sup>4,7</sup> In spite of satisfactory material

and technique, there has always been a never ending quest for newer and better materials and technique.

The application of laser technology to dentistry has opened many new avenues of applied research and clinical practice. A wide variety of lasers has been used in preventive and social dentistry, restorative dentistry, endodontics, periodontics and oral surgery.<sup>8</sup> Argon ion laser is tested and proved to polymerize restorative composite resins<sup>9,10</sup> and also shown to improve physical properties over conventional visible light curing.<sup>11-14</sup> Visible light cure (VLC) resin material shares same photo initiator system as composite resin. A comprehensive study was conducted to evaluate the applications of argon ion laser technology to VLC denture base resin polymerization.

The study was conducted in two phases.

- Phase 1: Finding out suitable exposure time required for optimum polymerization of trial VLC using argon ion laser.
- Phase 2: Comparing and evaluation the physical properties of conventional heat cured, light cured denture base material with argon laser polymerized VLC resin.

### Phase 1

The American Dental Association (ADA) specification No. 12 (ISO 1567, IS 6887)<sup>15,16</sup> for denture-base polymers include tests for working qualities, color stability, water sorption, water solubility, and transverse deflection. The flexural test is the only mechanical test listed, which is thought to be relevant since it reflects the loading arrangement in the clinical situation.

Denture base polymers may fail clinically because of flexural fatigue. It is therefore appropriate that flexural strength testing is used as a method for evaluating optimum polymerization. Since flexural properties may vary with specimen depth, support spans length, temperature, atmospheric conditions, and rate of straining. Testing procedures were carefully standardized as per ADA specification No. 12 for denture base polymers. According to ISO 1567 VLC resin (Type 4) should have minimum 65 MPa (6.628 kg/mm<sup>2</sup>) to qualify as denture base resin.<sup>16,17</sup>

### Materials and methods

*Triad visible light curing resin (Dentsply, York, PA)*

Triad material is similar to light-cured composites but uses organic rather than inorganic filler. The material is composed of a matrix of urethane di methacrylate plus small amounts of micro fine silica to control rheology. The filler consists of acrylic resin beads of varying sizes that become part of an interpenetrating polymer network structure when cured. Polymerization of high molecular weight acrylic resin monomers contained within the matrix is initiated with a

comphoro-quinone amine photo initiator. Air inhibition of the surface layer polymerization is prevented by applying an air-barrier coating before final polymerization. The material is manufactured in sheets and ropes and packaged in opaque plastic envelopes to prevent contamination by light. The system eliminates the need for the wax, flask, boil-out tanks, packing press and heat processing units required for conventional denture construction.<sup>5</sup>

The mould space for required specification size was prepared by using metal dies of size 65 mm × 10 mm × 2.5 mm, invested in Type 3 dental stone in a Hanau type denture flask. The triad VLC resin samples were prepared by adapting the material with finger pressure to dental stone mould. The specimens were carefully removed from the mould and coated with air barrier coating agent and kept in the path of argon laser (Coherent Innova 90) with a beam radius of 40 mm, the intensity was maintained at 1 W/mm<sup>2</sup>. The wave length was set at 488 nm.<sup>13</sup> The specimen were cured in two overlapping sections due to its length (65 mm). After lasing, the specimens were trimmed to the final dimensions i.e. 2.5 ± 0.03 mm depth, 10 ± 0.03 mm width and 65 ± 0.03 mm length with the help of digital caliper.

A total of 10 specimens were polymerized at 1 W/mm<sup>2</sup> power with exposure times of 3, 4, 5, 6, 7, 8, 9 and 10 min. Totally 80 specimens were prepared and stored in distilled water at 37°C for 48 ± 2 h before subjecting for testing. Immediately prior to testing, individual specimen were removed from water bath and dried using laboratory tissue. The three point transverse bending test was conducted on an Instron universal testing machine (model 6025, Instron Ltd., Wycombe, bucks, UK) at room temperature. The individual specimen was subjected to progressive loading with crosshead speed of 5 mm/min in compliance with ADA specification No. 12 for denture base polymers. Prior to three point loading, specimen alignment and simultaneous contact of the loading nose were checked. The applied loads were symmetrically distributed to avoid torsion, which raises the levels of stress in the beam.<sup>18</sup>

The values for flexural strength were calculated by the machine using the following equation,  $S = 3PL/2bd^2$

where  $P$  is the load at the fracture point expressed in Newton's,  $L$  is the distance between the supports (50 mm),  $b$  is the width of the specimen (10 mm), and  $d$  is the thickness of the specimen (2.5 mm).

The values were recorded in MPa and tabulated. Mean and standard deviation were calculated for every exposure time. Results were compared to established performance requirements set forth in the ADA specification for determining optimum polymerization.

## Results and Discussion

Today lasers are the center of investigation to the entire researcher in the field of dentistry. The use of argon laser for polymerization of photo cured composite resin is well established. The triad VLC material of similar composition to composite resin was tested successfully in the same line, because the argon laser wave length is within the range of 400-500 nm. The wave length selected for polymerizing the triad VLC was 488 nm.<sup>13,14</sup>

In the first phase of the study, the correct exposure time for optimum polymerization of triad with argon laser was found out, keeping ADA/ISO specification as the standard.

Ten sample each laser cured for 3, 4, 5, 6, 7, 8, 9 and 10 min exposure time with 1 W/mm<sup>2</sup>, totally 40 samples prepared. Table 1 shows flexural strength (mean value) of specimen cured for 3, 4, 5, 6, 7, 8, 9 and 10 min. It is seen that the specimen cured for 7, 8, 9, and 10 min satisfy ISO standards. The increase in strength from 3 to 10 min of curing may be attributed to better degree of polymerization. However, there was no much significant increase in value of 9 min and 10 min cured specimen when compared to specimen cured in 8 min. Hence, it was decided to consider 8 min as the suitable time for optimum polymerization with argon laser.

Wave length	488 nm
Type of wave	Continuous
Power intensity	1 W/mm <sup>2</sup>
Exposure time	8 min

## Phase 2

After establishing the possibility of argon ion laser curing of VLC resin and also the parameters to achieve the optimum polymerization, the argon lased specimens were compared with light cured and conventional heat cured specimen. These specimens were tested for three point transverse bend test, impact test, surface hardness and surface characteristics by scanning electron microscope (SEM) study.

## Materials and methods

### Conventional heat cured resin (Acralyn H, Asian acrylates)

Pre-weighed sachets of powder and liquid were used according to manufacturer recommended powder liquid ratio. The processing was done according to manufacturer's instruction. Rectangular metal dies measuring 65 mm × 10 mm × 2.5 mm were used to create uniform mold spaces. The specimens of heat cured material were produced in Type 3 gypsum molds following conventional laboratory procedures<sup>3</sup> as per the

manufacturer's instruction. Flasks were always allowed to bench cool before being deflasked.

After being deflasked individual specimens were trimmed in a conventional procedure to final specimen dimensions, 2.5 ± 0.03 mm depth, 10 ± 0.03 mm width and 65 ± 0.03 mm length. Totally 30 samples of heat polymerized specimens were prepared and stored in distilled water at 37°C for 48 ± 2 h before testing.

### VLC resin (Triad, Dentsply, York, PA)

The gypsum mold space was prepared as explained before in a denture flask. Triad VLC material was adapted with finger pressure to dental stone mold. The triad specimens were processed initially in the mold for 2 min then removed and processed on the tissue side for 8 min keeping in the triad light chamber total of 10 min curing.

Important component of the curing unit is the emission of intense collimated shielded light from quartz halogen lamps that are concentrated in the shorter blue range 400-500 nm wave length spectrum of visible light and 90 MW power at curing temperature of 165-170°F.<sup>5</sup> High intensity light results in deep polymerization of material to depth 5-6 mm. 30 specimens of specifications size were prepared and stored in distilled water at 37°C for 48 ± 2 h before testing.

### Argon ion laser cured resin

The specimens were prepared as explained in Phase I of the study with exposure time of 8 min to argon laser. Totally 30 samples prepared and stored in distilled water at 37°C for 48 ± 2 h before testing.

### Flexural strength

10 specimens were used from each polymerization method. The specimen is mounted on the loading bars placed 50 ± 0.1 mm apart with cross head speed of 5 mm/min. The specimen alignment and simultaneous contact of the loading bars were checked. The amount of load at fracture was recorded by the machine and calculates flexural strength of the specimens in MPa.

### Impact strength

10 un-notched specimens<sup>19</sup> of size 65 mm × 10 mm × 2.5 mm were used from each curing method and tested for flexural impact strength. Tests were carried out in charpy type pendulum impact tester (Dynatup, GRC 730-I, General Research Corp., CA). This instrument measures the energy

Table 1: Flexural strength at different curing time.

Flexural strength (MPa)	3 min	4 min	5 min	6 min	7 min	8 min	9 min	10 min
Mean	33.22	53.86	58.5	62.8	76.35	84.71	86.31	89.75
Standard deviation	0.503	0.246	0.385	0.382	1.213	0.577	0.247	0.773

required to fracture the specimen by recording the reduced swing and hence the reduced kinetic energy of the pendulum. The energy consumed and strain before fracture is recorded in the form of graphs plotted through the printer. The tests were carried out using a pendulum rated 15 J and specimen supports having 50 mm separation with a flat side facing the pendulum. The machine gives amount of energy consumed for breaking the specimen by the pendulum. The impact strength is calculated using the formula given below. The mean and standard deviations were calculated.

$$a = (J_1 - J_2) 10^3 / bh$$

where  $J_1$  is the value of energy in joules absorbed by the specimen strip,  $J_2$  is the friction energy in joules of the system,  $h$  is the height in mm of specimen and  $b$  is the depth in mm behind the notch. Impact strength is expressed in  $\text{KJ/M}^2$ .

#### Surface hardness test

Ten Specimens of same dimensions from each curing method were tested for surface hardness in micro hardness tester (Shimadzu, HMV 2000, Japan). A simple machine with a microscope attached to a video monitor. This machine can measure the dimension of indentations on the specimen made by the diamond indenter accurately through an optical micrometer. The inbuilt program of the machine conducts automatically the indentation at specified load and calculates the Vickers hardness number. A load of 10 g was used with triangular pyramid indenter. One indentation made in each specimen. Mean and Standard deviations calculated.

#### Surface characteristics

Surface details of the surface of light cured, heat cured and laser cured acrylic resin samples were examined under SEM (Jeol, JSM 840-A, Japan). The specimen were coated with approximately 400 Å of 24 karat gold. The Samples were studied under the SEM operating at 20 KV and distance of 39 mm.<sup>20</sup>

The high ( $\times 300$ ) power views of surface of each of the three materials were photographed. The fractured surfaces the laser cured and light cured resin were viewed at  $\times 200$  magnification. The protective plastic cover was also studied at  $\times 200$  magnification after peeling off the uncured VLC resin.

#### Results and discussion

In Phase II study laser cured specimens using the parameters set by Phase I study was compared with light cured and heat cured material for flexural strength, impact strength, surface hardness and surface characteristics (SEM study). Table 2 shows the mean values flexural strength (MPa), impact strength ( $\text{KJ/M}^2$ ) and surface hardness (VHN) of laser cured, light cured and heat cured specimens.

Data collected were entered in an Excel format and descriptive and analytical statistics were computed. The statistical analyses were done with the SPSS Version 16 software package.  $P < 0.05$  was considered as significant. The data were checked for normalcy (Shapiro–Wilk test) and accordingly parametric tests were used. Descriptive statistics with frequency mean and standard deviation was computed. One-way ANOVA was used to determine whether there are any significant differences between the means of three independent (unrelated) groups and Tukey *post-hoc* test were used to find differences between any two groups. Results for one-way ANOVA for flexural strength, impact strength and surface hardness showed there were statistically significant differences between group ( $P < 0.001$ ). Tukey *post-hoc* test confirmed statistically significant differences between any two groups. ( $P < 0.001$ ).

The light cured has higher flexural strength compared to heat cured and followed by laser cured specimens. The impact strength of heat cured resin was highest, followed by laser cured and light cured samples. The light cured material has higher surface hardness value than laser cured material, followed by heat cured specimens.

The surface of laser cured, light cured and heat cured were studied under SEM and Figures 1-3 show  $\times 300$  magnification views of the same materials, respectively. The study of fractured surfaces of heat cured material showed more dense and compact structure as compared to light cured and laser cured resin examined. At  $\times 300$  magnification

Table 2: Comparative values of physical properties.

Material	Flexural strength MPa	Impact strength $\text{KJ/M}^2$	Surface hardness VHN
Laser cure (mean $\pm$ SD)	84.71 $\pm$ 0.577	3.15 $\pm$ 0.543	24.08 $\pm$ 1.031
Light cure (mean $\pm$ SD)	102.41 $\pm$ 2.189	2.2 $\pm$ 0.539	30.59 $\pm$ 1.778
Heat cure (mean $\pm$ SD)	98.27 $\pm$ 3.226	6.15 $\pm$ 0.602	20.86 $\pm$ 1.296

SD: Standard deviation

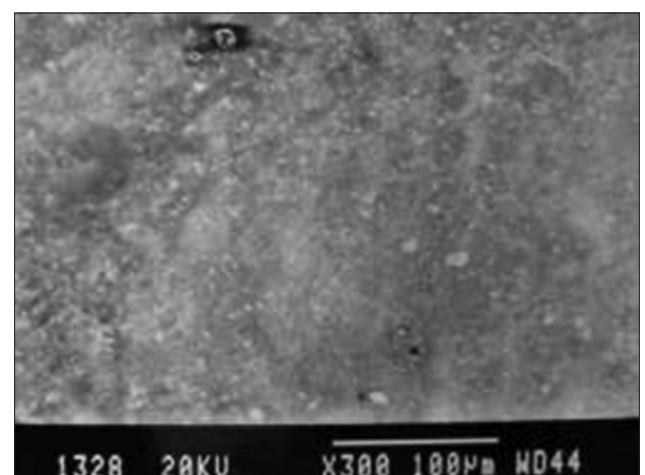
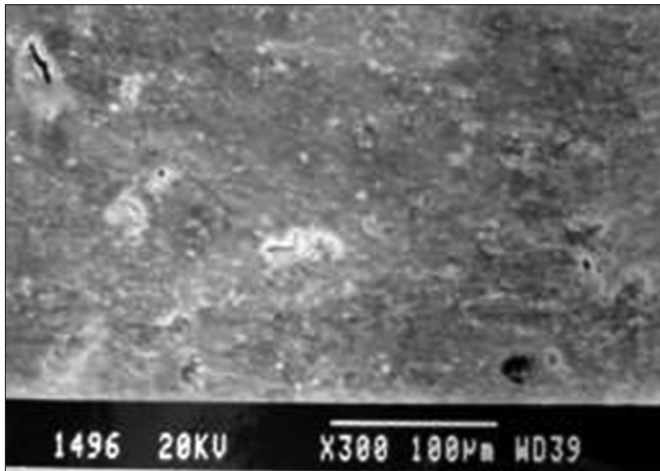


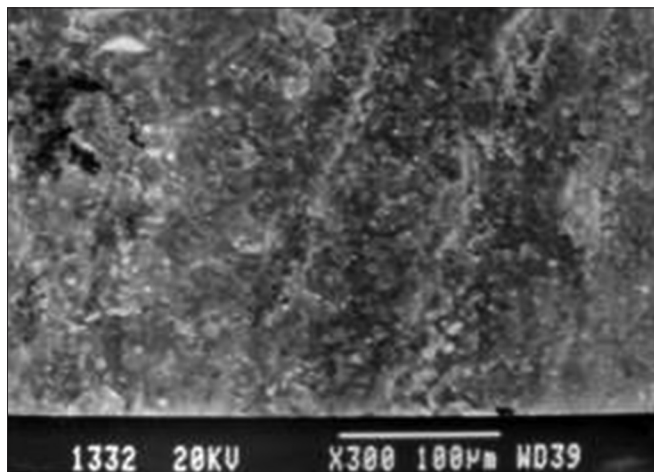
Figure 1: Heat cure resin surface at  $\times 300$  (1328).

Figures 4-6 shows the higher molecular weight resin bead filler particles, which are separated from the surface can be seen in laser cured and light cured resin materials.

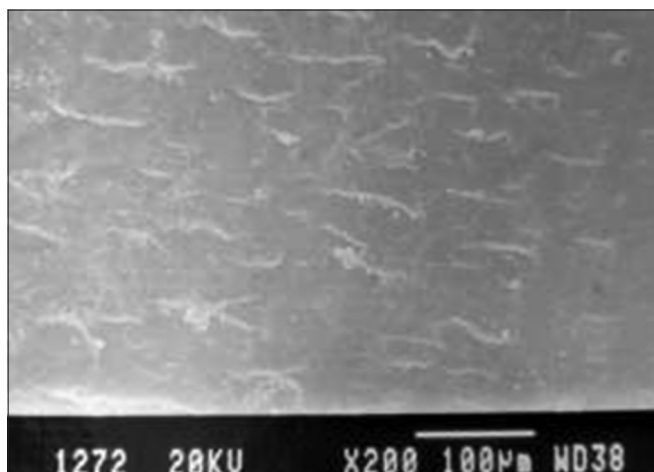
Denture base polymers may fail clinically because of flexural fatigue. It is therefore appropriate that flexural



**Figure 2:** Light cure resin surface at  $\times 300$  (1496).



**Figure 3:** Laser cure resin surface at  $\times 300$  (1332).

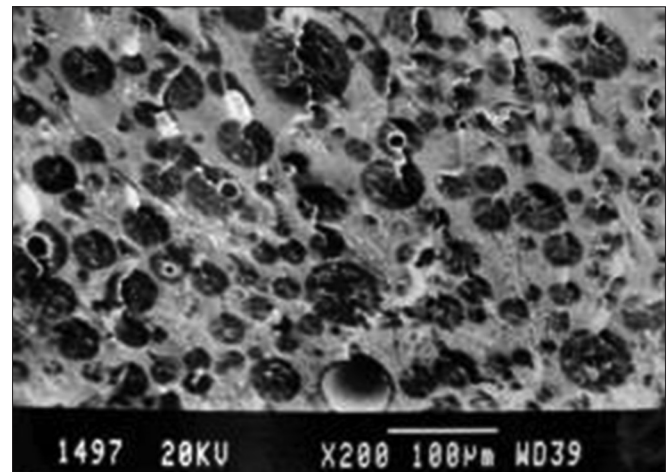


**Figure 4:** Heat cure resin fracture surface (1272).

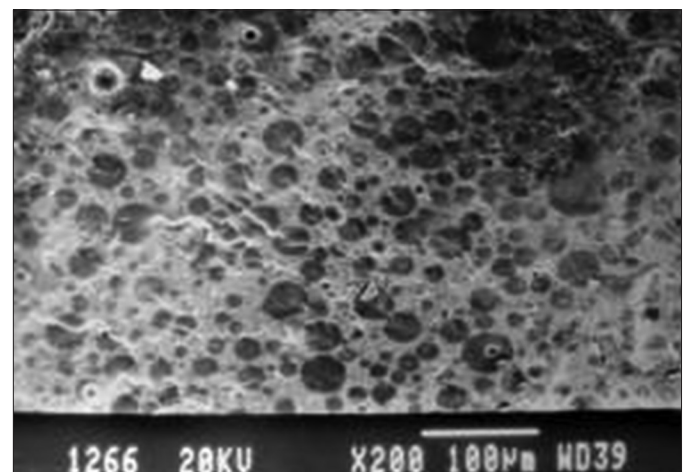
strength testing was used as a method of comparing the polymers performance.<sup>21,22</sup> Second, the flexural strength is very much sensitive to testing procedures and specimen dimensions.<sup>18</sup>

The study shows that the light cured samples had higher flexural strength, followed by heat cured conventional resin and laser cured VLC resin. The results for conventional heat cured and light cured resin are comparable with other studies.<sup>4,7,21,23-29</sup> The results of the present study indicate that the light cured denture base resin is more rigid than conventional heat cured and laser cured VLC denture base resin. This may be due to the presence of urethane dimethacrylate, which imparts stiffness to the resin.<sup>29</sup> However another *in-vitro* study conducted in water and artificial saliva showed lowest flexural strength of triad VLC compared to other conventional denture base resin.<sup>30</sup> These variations in test result we attributed to decomposition of particles in triad material following saturation in the water and saliva.<sup>31</sup>

The resistance to fracture of denture base polymer depends on one more factor, impact strength. The flexural strength



**Figure 5:** Light cure resin fracture surface (1497).



**Figure 6:** Laser cure resin fracture surface (1266).

gives an indication of material performance under conditions of static loading while impact strength involves testing under dynamic loading conditions. The unnotched specimens were used because of the notch sensitivity of the denture base polymers.<sup>19</sup>

The results indicated that impact strength of conventional heat cured resin was 3 times higher than light cured triad resin and 2 times higher than laser cured VLC resin. The impact values for the laser cured VLC resin was 1½ times higher than light cured specimen. This implies that light cured denture base resin has poor resistance to impact force as compared to conventional heat cured resin, however, curing VLC resin using argon laser resulted in slight improvement of the impact strength. Above results obtained for heat cured resin and light cured resin are in compliance with other studies.<sup>21,29,30</sup>

Surface hardness is the result of compactness of the molecules and the interaction of numerous properties, among the properties that influence the hardness of the material are its strength, proportional limit, ductility, malleability and resistance to abrasion and cutting. It is often been used as an index to resist abrasion.<sup>3,27</sup> Hence, Vickers hardness test was conducted.

The surface hardness of three materials was compared in a Vickers hardness tester. The light cured denture base resin had the highest hardness followed by laser cured VLC resin and conventional heat cured denture base resin. Hence, abrasion resistance of light cured and laser cured VLC resin material were better than the conventional heat cured resin<sup>21,28</sup> in compliance with other studies.

SEM study was conducted to evaluate the surface porosity and microscopic structure. This in turn gives an indication of strength.<sup>4</sup>

The SEM study of surface of specimens at ×300 magnification shows that the light cured resin surface appeared less dense and more irregular than the heat cure resin. The laser cured surface appeared almost similar to light cured resin in density and porosity. This change in the appearance of light cured and laser cured surface when compared to conventional heat cured resin, possibly because of the finger pressure adaptation technique used. Another reason for surface irregularity may have been bead separation. On initial observations of the VLC resin material, removal of the clear film coating after opening the light-safe package was found to result in separation some surface resin beads. These beads apparently adhered directly to the clear film as it was peeled away from the soft VLC resin surface and produced a mottled appearance on the surface cured. This can be seen on SEM study of plastic sheet. This finding is in good agreement with the study of Ogle *et al.*<sup>4</sup>

The study of fractured surfaces of heat cured material showed more dense and compact structure as compared to light cured and laser cured resin at ×200 magnification. The higher molecular weight resin bead filler particles, which are separated from the surface can be seen in laser cured and light cured resin materials.<sup>29</sup>

The study indicates argon laser can be used as an alternate to visible light in curing Triad material. The exposure time of argon laser to achieve required strength in the material is less than the exposure time advocated for light cured resin. A significant improvement in the impact strength was observed with the laser curing. The resistance to abrasion also better than heat cured resin. The SEM study indicated similar surface configuration for both light cured resin and laser cured VLC resin.

However, when the flexural strength and hardness was compared with light cured material there was slight decrease in the values of argon laser cured VLC resin. However, these values are comparable with the conventional heat cured resin. It may be difficult to explain the variation in the behavior of argon ion laser cured VLC resin and requires further study at molecular level for better reasoning.

Further study can also conducted at different power settings to find out any changes in the properties of laser cured VLC resin.

### Conclusion

The conclusions drawn from the above study as follows:

1. Triad VLC resin can also be polymerized with argon ion laser
2. Optimum polymerization of VLC resin with argon laser was achieved at 1 W power intensity and exposure time of 8 min
3. Impact strength of laser cured resin was better than light cured resin
4. Surface hardness of laser cured resin was better than conventional heat cured resin
5. Surface characteristic of laser cured VLC resin was comparable to surface characteristic of light cured resin
6. The flexural strength of laser cured VLC resin was slightly less than the flexural strength of light cured resin but meets ISO standards.

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