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Flexural Strength and Degree of Polymerization of a Proprietary Denture Base Acrylic Resin Designed to be Cured using Long or Short Cycles

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Abstract - *The flexural strength and degree of polymerization of Diamond D acrylic resin prepared with a long cure monomer and a short cure monomer were investigated using Trevalon as a control. Flexural strength and degree of polymerization of Diamond D acrylic resin were not affected by either using a long cure monomer or the short cure monomer. There was no significant difference in the glass transition temperature Tg between the long and slow cure Diamond D. Provided the manufacturer's instructions are followed the flexural strength, degree of polymerization and glass transition temperature are comparable with more traditional products.*

KEYWORDS: Poly(methylmethacrylate), denture base, processing of denture bases

INTRODUCTION

Since its introduction in the nineteen thirties acrylic resin denture base materials have been continually developed by their manufacturers. Problems encountered have been related to the curing cycle used in the production of dentures. The time and temperature employed have been important variables, which have been shown to affect the level of residual monomer[1,2,3], flexural strength[4,5] and possibly porosity of the resultant denture base[6].

Yau et al[6] showed that provided the clamp pressure exceeded the pressure in the mould developed by the bench press used to close the flasks, the rise in pressure inside the mould rose to 22 ATM at which point the boiling point of monomer would be an unattainable 228°C, thus porosity would not occur. Clark et al[7] observed that when the internal temperature reached 72°C the dough was still rubbery. However, one hour later it had gone hard suggesting it had polymerised. They correlated these observations with those of Yau et al[6] and hypothesised that as the temperature rose to 72°C thermal expansion caused the pressure in the dough to rise. The rate of polymerisation would increase when the temperature rose above 60°C and the initiator begins to dissociate. Once the temperature stops rising, polymerisation contraction would replace thermal expansion and the pressure would drop. They suggested that when the pressure had reached its minimum, polymerisation contraction had also reached its minimum and polymerisation was for practical purposes complete. On this basis they suggested that manufacturers' processing instructions for shorter cycles could safely be followed.

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Athar et al[8] investigated the effect that different curing cycles had on the mechanical properties of three brands of heat cured acrylic resin denture base material (Trevalon C, Meadway supercure, Paladon 65). They concluded that specimens achieved the British Standard specification with both short and long curing cycles and concluded that manufacturers' recommended curing cycles should be followed.

Recently a high impact heat cured acrylic resin, Diamond D Heat Cure Acrylic Resin, which has the option of both long and short cure monomers has been introduced. However, the possibility that the shorter curing cycle may adversely affect the mechanical properties of the polymer with the potential of higher amounts of residual monomer in comparison to the conventional curing monomer needs to be investigated.

The purpose of this study therefore was to compare Diamond D ultra impact denture acrylic, cured using Diamond D acrylic resin with long cure monomer (LCDD) and Diamond D acrylic resin with short cure monomer (SCDD) with a widely used product Selectaplus H/ Trevalon/ Universal Clear (TC) to investigate if the curing cycle affects the quality of these processed resins.

Two null hypotheses were tested: there is no statistically significant difference in the flexural strength between the short cure and the conventionally cured Diamond D acrylic resins and Trevalon (control) and there is no statistically significant difference in the degree of polymerisation of the three processed acrylic resins.

MATERIALS AND METHODS

Specimen preparation

Twelve standard specimens (BS EN ISO 1567: 2001) 50 ± 0.2 mm long, 10 ± 0.2 mm wide and 3.3 ± 0.2 mm in thickness were made using each of the three different materials (LCDD, SCDD and TC) following the manufac-

turers' recommended mixing and curing instructions. The liquid/powder ratio for SCDD was 10 ml monomer to 21g powder, whereas for LCDD and TC was 10 ml monomer to 22g polymer. The recommended amount of each monomer was measured with a graduated syringe and poured into a clean glass mixing jar with lid. The polymer containers were shaken for 30 seconds to distribute the polymer powder and fibres evenly. Using a stainless steel spatula, a pre-weighed (42g from the SCDD, 44g from the LCDD and TC) powder was stirred gradually into the monomer until the monomer was completely taken up. The jar was tapped on the bench top to bring the remaining monomer to the surface and the remaining powder was added. Stirring was continued until the entire polymer had been wetted. The jar was covered and left for 10 minutes at room temperature 20 ± 2°C to reach the dough stage when it pulled away from the sides of the jar and did not adhere to the stainless steel spatula.

Processing cycles recommended by their manufacturers' were used as follows. The flasks containing SCDD were immersed in room temperature water (20 ± 2°C) and brought up to 100°C and maintained for 20 minutes. They were removed from the water and were bench cooled to room temperature for another 30 minutes before de-flasking. The flasks containing LCDD were placed in room temperature water (20 ± 2°C), slowly raised to 70°C in an automatically controlled water bath and maintained at that temperature for 4 hours. They were removed and bench cooled to room temperature for 20 minutes before de-flasking.

The flasks containing TC were immersed in room temperature water (20 ± 2°C), slowly raised to 70°C in an automatically controlled water bath and maintained at that temperature for 4 hours. They were removed and bench cooled to room temperature for 20 minutes before de-flasking.

The acrylic resin specimens were deflasked. The specimens were carefully removed and cleaned under running water. Only sharp fine edges were removed cautiously by finger pressure to prevent the build up of stresses within the specimens. The edges were refined with sandpaper taking care not to change the required dimensions (50 ± 0.2 mm long, 10 ± 0.2 mm wide and 3.3 ± 0.2 mm thick) according to the British Standards Specifications (BS EN ISO 1567: 2001).

Flexural strength

Ten specimens were subjected to three point bend tests (flexural strength) carried out according to the British Standard Specification (BS EN ISO 1567: 2001) using a Universal Testing Machine (Instron 1193). The distance between the two supports was 20mm and the plunger was centred in the middle and a crosshead speed of 1mm/min was used for testing. The flexural strength was calculated using the standard equation for a three point bend test: $Fl/4W_b$ where F=the fracture load and l= the distance between supports. For rectangular cross sections $W = bh^2/6$ where b = the width of the specimen and h = thickness. ANOVA was used to determine significant differences.

Fourier Transform Infrared spectroscopy (FTIR):

Non-polymerized monomer from each brand of the three heat cure acrylic denture bases was tested. The FTIR spectra were recorded using a Perkin Elmer FTIR spectrometer using a potassium bromide (KBr) disc, and each spectrum was obtained from 50 scans. A smear of the monomer was applied to the KBr disc which was then scanned in the spectrometer. The two remaining polymerised specimens were ground to fine shavings using a tungsten carbide bur and used for spectral determination. Spectra were smoothened and normalized using an automated baseline technique. The ratio of absorbance intensities C=C/C=O was compared before and after polymerisation to determine the percentage of un-reacted monomer. The degree of cure (DC%) was calculated from the aliphatic C=C peak at 1638 cm^{-1}, and normalized against the aromatic C=O peak at 1730 cm^{-1} according to the Equation 1.

Equation 1

$$\text{Degree of cure} = \frac{[\text{Abs (C=C)/ Abs (C=O)}]\ \text{polymer}}{[\text{Abs (C=C)/ Abs (C=O)}]\ \text{monomer}} \times 100$$

Differential Scanning Calorimetry (DSC)

The glass transition temperatures of the cured denture base polymers were determined using a Perkin Elmer jade DSC system. The system was calibrated with zinc/indium. Approximately 10µg of sample material were tested in crimpled aluminium pans at a rate of 10°C/min^{-1} under nitrogen gas (20 ml/min^{-1}) over a two-step heating/cooling cycle temperature range of -20 to 200°C. The glass transition was determined by the midpoint of the initial transition slopes using Pyris computer software.

RESULTS

Three point flexural strength: TC had the largest range of values and expressed the highest three point flexural strength 16.6 MPa; it also gave the lowest measurement 11.5 MPa amongst the three groups. Three point flexural strengths of SCDD ranged from 12.5 MPa to 13.9 MPa, which were similar to those of the LCDD 12.7 MPa to 14.5 MPa. Specimen 9 of TC fractured at 11.5 MPa and specimen 2 reached 16.6 MPa before it failed. This led to a wide standard deviation higher than that of the other two groups (Table1).

ANOVA showed the differences in the mean values among the treatment groups were not great enough to exclude the possibility that the difference is due to random sampling variability; there was no statistically significant difference ($p = 0.15$).

Table 1. *Descriptive statistics for the flexural strength of the three groups*

Group Name	*N*	*Missing*	*Mean*	*Std Dev*
Short cure D D	10	0	13.1	0.44
Long cure D D	10	0	13.6	0.62
Trevalon C	10	0	14.0	1.53

FTIR spectra:

Figure 1 shows the FTIR spectra of the three monomers of the denture bases used in this study. The FTIR spectra indicate that the monomers were similar with some differences in the chemical composition. As they are proprietary materials it is difficult to obtain their exact composition but a comparison of the FTIR spectra show very similar components present within the formulation, it being mainly methylmethacrylate.

A sharp intense peak centred at 1717cm^{-1} in the spectra arises due to the stretching vibration of the ester carbonyl group present in methylmethacrylate, the main component of the liquid phase. A sharp but lower intensity peak is observed at 1637cm^{-1} for all the monomers present in TC, LCDD and SCDD monomer due to the C=C stretching vibration. The peaks ranging from 1300-1000cm^{-1} can be associated with the C-O ester stretching vibration whilst the peaks between 960-650cm^{-1} arise from the –C-H bending modes. An additional peak at 1438cm^{-1} with higher intensity and a shoulder at 1453cm^{-1} was observed for SCDD monomer indicating differences in the composition.

The FTIR spectra of the polymerized specimens are shown in Figure 2. The absence or very low intensity peaks at 1637cm^{-1} shows high degree of conversion of all the three denture base polymers.

The FTIR spectra in Figure 2 also shows well defined peaks in the 1722 cm^{-1} which represents a carbonyl group, but the carbon-carbon double bond peak which is usually seen in the range 1636-1648 cm^{-1} wavelengths had nearly disappeared confirming that the material had been polymerized. The degree of polymerization and the residual carbon double bonds as determined by infrared spectroscopy are shown in Table 2 as percentages. LCDD showed the highest degree of monomer conversion to polymer (99.8%), followed by SCDD (99.7%) and TC acrylic resin (99.6%).

Differential Scanning Calorimetry

The glass transition temperature of TC, SCDD and LCDD are shown in Table 3, which is an average of two measurements for each. There is no significant difference between the Tg of the two types of Diamond D material, which have a similar composition and the values are comparable to literature values. This indicates as also shown by FTIR measurements that the degree of cure is similar in all the three systems and a shorter processing time did not adversely the Tg values.

DISCUSSION

Processing methods of heat cured acrylic resins used to fabricate denture bases vary and the resultant properties depend on the processing parameters. Low residual monomer content is important to impart optimum physical and biological properties. The short curing cycle for Diamond D acrylic resin is short even in comparison to other processing methods, thus it is important to determine the effects of a short cure in assessing the material properties. Both SCDD and LCDD acrylic resins were chosen for this study as there is no research into strength differences between the two cured resins. Trevalon C was selected as the control/contrast owing to its commercial popularity and widespread use and availability.

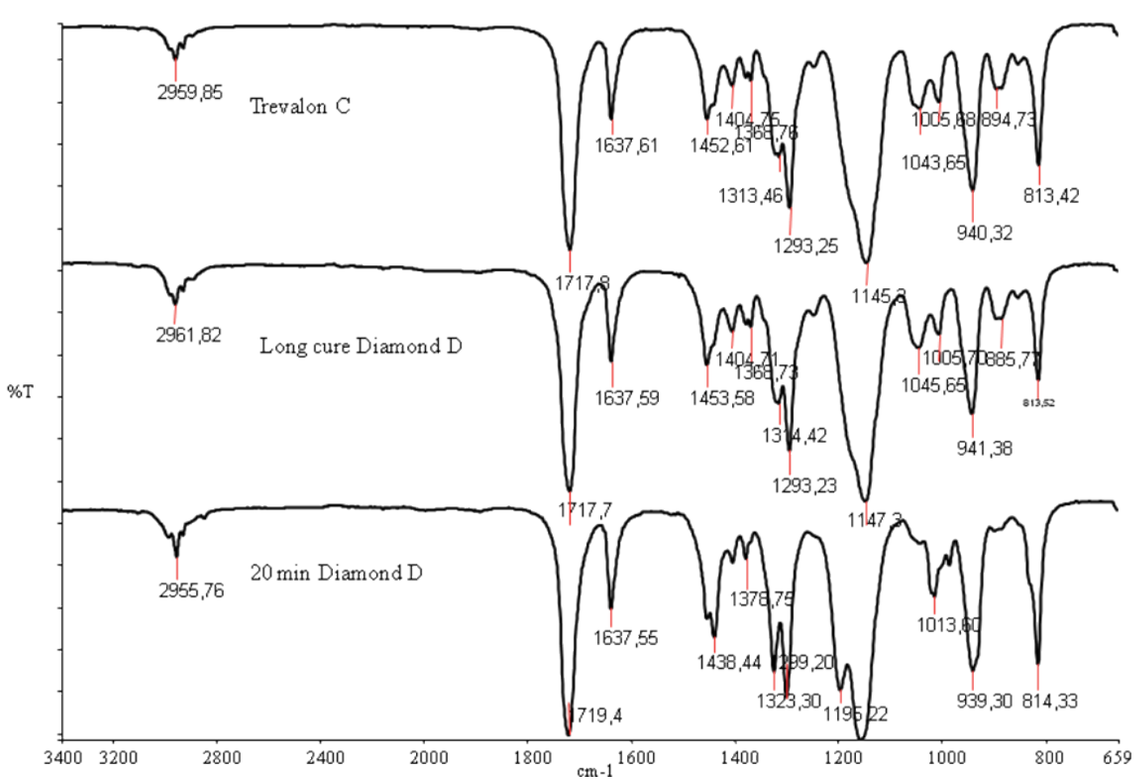

Figure 1. *FTIR spectra of the three monomers for Short cure(20 min) Diamond D, Long cure Diamond D and Trevalon C*

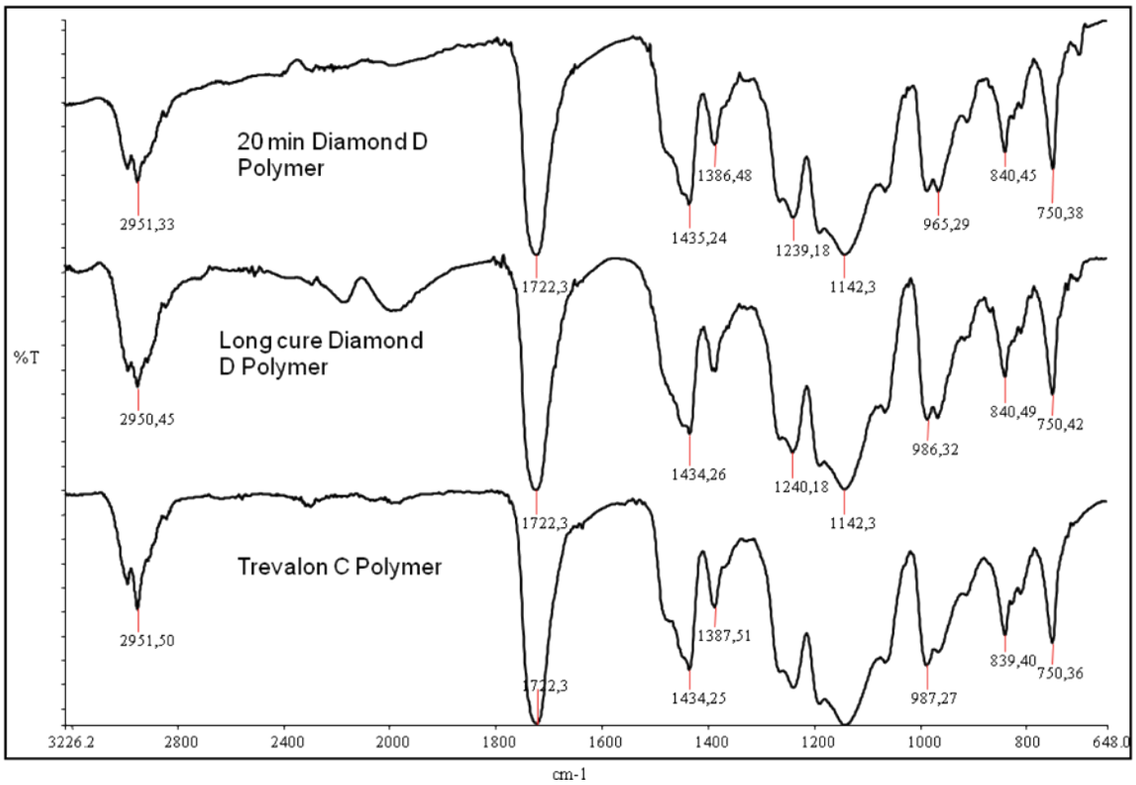

Figure 2. *FTIR spectra of the three polymerized denture base resins*

Table 2. *Degree of polymerization and residual carbon double bonds*

Type of acrylic resin	*Degree of polymerization (%)*
Short Cure Diamond D Acrylic Resin	99.7%
Long Cure Diamond D Acrylic Resin	99.8%
Trevalon C Acrylic Resin	99.6%

Table 3. *Glass transition temperature of the denture base polymers*

Type of acrylic resin	*Tg in ^{0}C*
Short Cure Diamond D Acrylic Resin	117.5
Long Cure Diamond D Acrylic Resin	118.3
Trevalon C Acrylic Resin	127.7

Flexural strength was tested as a measure of one of the most important mechanical properties of denture base acrylic resin. This investigation was chosen as it is considered to imitate similar loads that affect the denture in situ[10].

There was no statistical significant difference ($p = 0.15$) in the flexural strength between the three tested denture base acrylic resins. Therefore the fact that different curing regimes had no effect on mechanical properties of the three resins demonstrated that they were appropriate for each manufacturer's formulation. The FTIR measurements also indicated that degree of cure was comparable for the short curing cycle with the long cure Diamond D and Trevalon C. Tg is directly influenced by the amount of residual monomer[9]. There was no significant difference between the short and long cure time for Diamond D, however Trevalon C exhibited higher Tg.

Practical significance:

As far as failure in denture bases is concerned, it was concluded that SCDD acrylic resin has similar flexural strength as the LCDD and TC acrylic resins. Flexural strength and degree of polymerization of SCDD were not affected by the short curing cycle. Furthermore, the FTIR spectra showed that some alteration has been made to the monomer of SCDD to allow a short curing time cycle. This is in agreement with the views of Clark et al[7]. All findings in this present study were of practical significance, as they verified the manufacturer's recommendation that SCDD acrylic resin are reliable denture base resins which save time, by providing a quicker processing cycle, high degree of polymerization; with subsequently a very small amount of residual monomer and finally a good flexural strength. These results support the previously published view[6,7,8,] that the manufacturers' instructions should be followed.

CONCLUSION

Flexural strength, degree of polymerisation of Diamond D acrylic resin and the Tg were not affected by using either a long cure monomer or the short cure monomer. Therefore both the null hypotheses tested stand.

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MANUFACTURERS DETAILS

- Keystone Industries, Cherry Hill, NJ 08002, USA
- Dentsply Caulk, Milford, UK.
- Coated Abrasives, Standard Glasspaper 121, Grade M2, Grit 70
- Perkin Elmer Precisely, Spectrum One, FT-IR Spectrometer
- Instron, High Wycombe, UK
- Pyrus software

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