TIME-DEPENDENT DEFORMATION OF POLYMER-BASED PROVISIONAL CROWN AND FIXED PARTIAL DENTURE MATERIALS

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Statement of problem. One of the common problems of provisional crown and fixed partial denture materials is that when they are subjected to constant loads for a long period of time, they exhibit a dimensional change (creep).

Purpose. The aim of this study was to investigate the viscoelastic behaviour of polymer-based provisional crown and fixed partial denture materials with time at constant compressive load.

Material and methods. Three dimethacrylate-based materials (Protemp 3 Garant, Temphase, Luxatemp) and one monomethacrylate-based material (Trim) were selected. Dimensional changes of the specimens were recorded by a LVDT to evaluate their viscoelastic behavior and creep strain. For all specimens, two loading procedures were used. At first, static compressive stress of 4 MPa was applied for 30 minutes and followed by 1 hour of strain recovery. Then, after 24 hours of water storage, the specimens were loaded again. The creep values between materials were statistically analyzed using one-way ANOVA and multiple comparison Scheffé test. Independent samples t-test was also used to identify the difference of creep strain between first and secondary loading conditions at the significance level of 0.05.

Results. Following application of the first loading, Trim showed the highest maximum creep strain (32.7%) followed by Luxatemp, Protemp 3 Garant and Temphase, with values of 3.78%, 2.86% and 1.77%, respectively. Trim was significantly different from other materials ($P<0.05$), while there were no significant differences among Luxatemp, Protemp 3 Garant and Temphase ($P>0.05$). The highest recovery and permanent set of Trim, were significantly different from those of others ($P<0.05$). At the secondary loading of the dimethacrylate-based materials, creep deformation, recovery and permanent set decreased and the percentage of recovery increased, while in Trim, all values of the measurements increased. This result showed that the secondary loading at 24 hours produced a significant creep magnitude.

Conclusion. The dimethacrylate-based provisional crown and fixed partial denture materials showed significantly higher creep resistance and lower deformation than the monomethacrylate-based material. Thus, monomethacrylate-based materials should not be used in long-term stress-bearing situations.

Key Words

Creep, Polymer-based provisional crown and fixed partial denture material, Viscoelasticity

※ This research was supported by the Ewha Womans University Research Grant of 2004.

All dental prostheses should be strong enough to resist any change of shape when subjected to heavy masticatory load during function. Deformation behaviour is an important aspect to consider when selecting materials for provisional or permanent dental prostheses. There are two time-dependent phenomena. One is *fatigue.* When a material is subjected to constant or repeated load below the failure stress, the material will eventually fail. This is called dynamic fatigue. The effect of cyclic stress is to initiate microcracks at the centers of stress concentration or on the surface. Propagation of cracks leads to failure. The other is *viscoelasticity.* Viscoelastic materials show both elastic and viscous deformation behaviour. These viscoelastic properties are a significant aspect of the mechanical performance of polymers.

Elastic deformation behaviour is effective instantaneously. The total deformation occurs the instant the stress is applied and completely disappears the instant the stress is released. A perfectly elastic material stores all of the energy created by deformation stresses so that on removal of the stress, it can return to its original dimension. Viscous deformation behaviour is delayed in response to stress. In a perfectly viscous fluid, the stress created by external forces relaxes instantaneously to zero due to flow. This deformation is not fully reversible or completely recovered. Applied stress on polymers causes an instantaneous elastic strain, followed by a viscous, time-dependent strain.

Measurements that characterize material performance under constant strain or stress conditions can be classified into *stress relaxation* and *creep.*[1] These measurements can provide important information as to material properties under long term conditions. Stress relaxation measurements are usually performed under constant strain conditions. This usually involves going to a specific load or strain point then holding the strain value. The resulting decrease in load or stress values is recorded over time. On the contrary, creep measurements are usually performed under constant load or stress conditions. These types of measurements are performed by going to a specific load or stress point, then holding the load or stress value. The resulting increase in strain is recorded over time.

Polymer-based provisional crown and fixed partial denture (FPD) restorations are subjected to load by mastication. It is important that there should be no change in their dimension during function. However, when polymers are subjected to constant loads for a long period of time, they exhibit a dimensional change (creep). This is the slow progressive, time-dependent deformation resulting from application of constant stress to a material when the stress is below the material' s yield stress. This is one aspect of their viscoelastic nature, and information concerning the polymers is important as a guide to service performance. Thus, creep is an important factor in the mechanical performance of polymer-based provisional crown and FPD materials.

The objectives of this study were (i) to investigate the viscoelastic behaviour of polymer-based provisional crown and FPD materials with time at constant compressive load, and (ii) to investigate the difference in creep strain between the first and the secondary loading. The null hypothesis to be tested was that there was no difference in the creep strain between monomethacrylate-based and dimethacrylate-based provisional crown and FPD materials.

MATERIALS AND METHODS

Four polymer-based provisional crown and FPD materials used in this study are presented in

Table I. Protemp 3 Garant (PT3), Temphase (TMP), Luxatemp (LXT), which are two paste systems, are dimethacrylate-based resins. Trim (TRM), which is a powder/liquid system, is a monomethacrylate-based resin. All four materials were chemically-activated resins. All materials were automatically mixed by a dispenser tip except for TRM which was mixed manually according to manufacturer' s instructions.

Cylindrical specimens of each material were prepared in a stainless steel mold which can be split so that no force was required to remove the set material. The mold had a cylindrical hole, 6 mm in length and 4 mm in diameter. The mixed materials were placed into the mold by the automix gun, while TRM was delivered by a plastic spatula. The mold was overfilled by the mixed material with great care to minimize the entrapment of porosity. A glass slab with a plastic matrix film was placed onto the mold to prevent the inhibition of polymerization by oxygen and to create flat end surfaces, and then hand pressure was applied. The specimen was left to polymerize for 30 minutes at 23℃ ± 1℃. After the material was set, the mold was disassembled, and the specimen was removed from the mold. The excess flash of the specimen was carefully trimmed by abrading with 800 grit sandpaper under running water to ensure that both end surfaces were perpendicular to the long axis of the specimen. Three specimens were fabricated for each material. Each specimen was placed into a small labelled bottle with distilled water. The bottles containing the specimens were stored in an oven at 37℃ for 2 hours prior to testing.

A creep testing apparatus used in this study consisted of a lever arm, which pivoted at one end *via* a bearing pin in a vertical pillar bolted to a steel U section base. A loading pin of 10 mm diameter, which was contained in a reduced friction bearing, was located vertically. The pin was displaced linearly by the angular motion of the lever arm. The specimen was placed on a platform in axial alignment with the loading pin. The platform was located in a small water bath where water of 37℃ was circulated during the experiment for the purpose of temperature control. The load was applied symmetrically along the specimen axis.

Table I. The polymer-based provisional crown and fixed partial denture materials investigated

Material	Code	Shade	Manufacturer	Characteristics
Protemp 3 Garant	PT3	A3	3M-ESPE, St. Paul, Minnesota, USA	Two component system (catalyst & base) based on multifunctional methacrylate esters, dimethacrylate (bis-acrylic)-based composite.
Temphase	TMP	A3.5, fast set	Kerr, Orange, California, USA	Two component system, 41% filled. particle size : 0.6 mm, dimethacrylate-based composite.
Luxatemp	LXT	A2	DMG, Hamburg, Germany	Two component system, multifunctional methyl-acrylic material. Bis-GMA composite resin formulation, Dimethacrylate-based composite.
Trim	TRM	Dark	Bosworth, Skokie, Illinois, USA	Powder/liquid system, monomethacrylate-based material, vinylethyl methacrylate

Two loading procedures were used for all specimens. Firstly, a static compressive stress of 4 MPa was applied to the end surface of each specimen by the application of weight to the free end of the lever arm, and maintained for 30 minutes. This was followed by 1 hour of strain recovery. After the first test, the tested specimen was stored in the bottle with distilled water of 37°C prior to the next experiment. Secondly, after water storage up to 24 hours after completion of mixing, the specimen was loaded again by the same compressive stress of 4 MPa for 30 minutes, and then the load was removed for 1 hour.

Dimensional changes of the specimens were monitored in the units of voltage by the LVDT (Linear Variable Displacement Transducer, Type GT 2000, RDP Electronics Ltd., Wolverhampton, UK), which was resting on an outrigger clamped to the loading pin. A signal from the LVDT was transferred to an analogue to digital converter (Pico Technology Ltd., Hardwick, Cambridge, UK), and recorded by a computer data recorder (*DASY Lab* software, Version 5.02, DATALOG GmbH & Co. KG, Moenchengladbach, Germany). With the aid of *Fig.P* (The Scientific Fig. Processor, Version 2.98, *Fig.P* Software Corporation, Durham, NC, USA), the dimensional change ($\Delta L = L_o - L$, where L_o is the original specimen length and L is the final length) was determined *via* the calibration coefficient of displacement/voltage. This change (ΔL) was divided by 6mm (L_o), and then multiplied by 100 to get a percentage of dimensional change as a function of time.

In addition, the length of the specimen was also measured with a digital caliper before and after each test, and its value was recorded. Three specimens per material were tested under the identical conditions.

The displacement/voltage calibration factor was calculated by linear regression. The calibration coefficient was 4.45×10^5 mm/mV ($R^2 = 0.99$). The transducer was calibrated periodically throughout the course of the work.

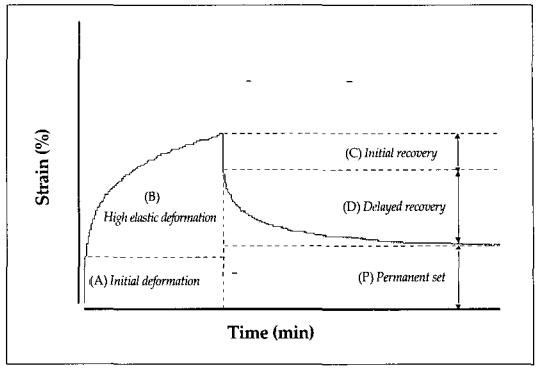

Fig. 1. Standard deformation curve subjected to load application and removal.
(A): Instant rapid elastic deformation, (B): Visco-elastic deformation, (C): Instant recovery, (D): Visco-elastic delayed recovery, (P): Permanent set.

An idealized time-dependent deformation curve subjected to constant stress is illustrated in Figure 1. When a polymer is loaded below its elastic limit, there is an instant rapid elastic deformation ***(A)***. This is followed by a slower time dependent, viscoelastic deformation ***(B)*** which is known as creep. When the load is removed, an instant recovery takes place ***(C)***. This is also followed by a further time-dependent, viscoelastic recovery ***(D)*** that may or may not complete with the given time limit of a test, and then by a permanent set ***(P)***.

In this experiment, the following measurements were derived:

(1) Instant rapid elastic deformation *(A)*
(2) Viscoelastic deformation *(B)*
(3) Maximum creep strain ($Y_1 = A + B$)
(4) Instant and delayed recovery ($Y_2 = C + D$)
(5) Permanent set *(P)*
(6) Percentage of creep recovery ($R = [Y_2/Y_1]\ H\ 100$)

The mean values and standard deviations of the results were computed. All data were statistically analysed by independent samples t-test at the significance level of 0.05. One-way ANOVA

and the multiple comparison Scheffé test were also used at the significance level of 0.05. SPSS software (Version 11.0, SPSS Inc., Chicago, Illinois, USA) was used for these statistical analyses.

RESULTS

The mean values and the standard deviations of creep deformation are shown in Table II and illustrated graphically in Fig. 2. The statistical analyses are presented in Tables III to V.

The First Loading

The kinetic curves of creep strain vs. time of different materials were all similar with the exception that the curves for different materials were shifted higher or lower on the creep scale.

Following application of the compressive load, all of the materials deformed with rapid instantaneous deformation *(A)* by 0.45% - 1.08%. LXT showed the highest initial deformation. It was followed by a slower, decelerating viscoelastic creep phase *(B)*. TRM had the highest value by 31.7 %. Continued loading resulted in a constant rate of creep until the load was removed at 30 minutes. TRM showed the highest maximum creep strain *(A + B)* (32.7%) followed by LXT, PT3 and TMP, with values of 3.78%, 2.86% and 1.77% respectively. TRM was significantly different from other materials ($P<0.001$), while there were no significant dif-

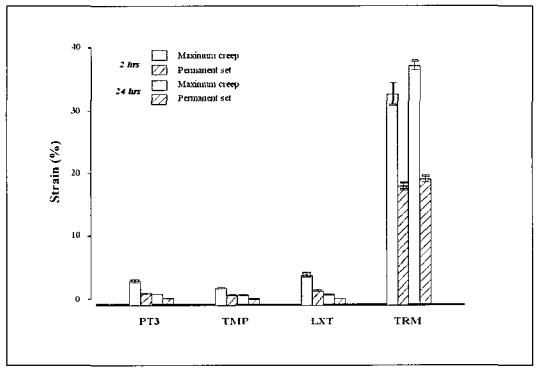

Fig. 2. Maximum creep and permanent set of provisional fixed partial denture materials investigated.

Table II. Mean values and standard deviation in parenthesis of creep deformation measured by LVDT

Material	Time	Max. strain (%)	Permanent set (%)	Recovery (%)	*A* (%)	*B* (%)	Y_1 (%)	Y_2 (%)	*P* (%)	*R* (%)
PT3	2h	2.86 (0.15)	0.99 (0.07)	65.5 (1.44)	0.65 (0.07)	2.21 (0.19)	2.86 (0.15)	1.87 (0.11)	0.99 (0.07)	65.5 (1.44)
	24h	0.84 (0.02)	0.16 (0.01)	81.3 (1.78)	0.43 (0.02)	0.41 (0.00)	0.84 (0.02)	0.68 (0.03)	0.16 (0.01)	81.3 (1.78)
TMP	2h	1.77 (0.08)	0.65 (0.05)	63.4 (1.03)	0.45 (0.21)	1.32 (0.26)	1.77 (0.08)	1.12 (0.03)	0.65 (0.05)	63.4 (1.03)
	24h	0.69 (0.10)	0.13 (0.05)	81.5 (6.25)	0.44 (0.19)	0.25 (0.10)	0.69 (0.10)	0.56 (0.08)	0.13 (0.05)	81.5 (6.25)
LXT	2h	3.78 (0.30)	1.39 (0.16)	63.4 (1.33)	1.08 (0.13)	2.70 (0.18)	3.78 (0.30)	2.39 (0.14)	1.39 (0.16)	63.4 (1.33)
	24h	0.84 (0.08)	0.13 (0.02)	84.6 (1.03)	0.44 (0.09)	0.4 (0.02)	0.84 (0.08)	0.71 (0.06)	0.13 (0.02)	84.6 (1.03)
TRM	2h	32.7 (1.70)	18.0 (0.57)	44.9 (1.90)	1.01 (0.04)	31.7 (1.72)	32.7 (1.70)	14.7 (1.35)	18.0 (0.57)	44.9 (1.90)
	24h	37.2 (0.75)	19.2 (0.50)	48.4 (0.80)	1.20 (0.81)	36.0 (0.78)	37.2 (0.75)	18.0 (0.46)	19.2 (0.50)	48.4 (0.80)

Table III. One-way ANOVA test of creep values between materials

Time	Data	DF	Sum of squares	Mean square	F	Sig
2h	*A*	3	804	0.268	16.195	
	B	3	1980.1	660.033	849.774	<0.001
	Y_1	3	2021.954	673.985	892.429	<0.001
	Y_2	3	371.418	123.806	248.540	<0.001
	P	3	653.115	217.705	2455.550	<0.001
	R	3	839.950	279.970	131.236	<0.001
24h	*A*	3	1.304	0.435	2.462	0.137
	B	3	2864.619	954.873	6236.245	<0.001
	Y_1	3	2988.126	996.042	6849.573	<0.001
	Y_2	3	677.167	225.722	4099.072	<0.001
	P	3	820.346	273.449	4246.540	<0.001
	R	3	2641.980	880.660	80.127	<0.001

Table IV. Multiple comparisons Scheffé test of creep values

Time	Material	*A*	*B*	Y_1	Y_2	*P*	*R*
2h	PT3 vs. TMP	0.380	0.684	0.534	0.653	0.604	0.453
	PT3 vs. LXT	0.023	0.922	0.654	0.842	0.481	0.428
	PT3 vs. TRM	0.050	<0.001	<0.001	<0.001	<0.001	<0.001
	TMP vs. LXT	0.003	0.358	0.118	0.258	0.090	1
	TMP vs. TRM	0.005	<0.001	<0.001	<0.001	<0.001	<0.001
	LXT vs. TRM	0.945	<0.001	<0.001	<0.001	<0.001	<0.001
24h	PT3 vs. TMP	1	0.965	0.970	0.934	0.999	1
	PT3 vs. LXT	1	1	1	0.999	0.999	0.695
	PT3 vs. TRM	0.248	<0.001	<0.001	<0.001	<0.001	<0.001
	TMP vs. LXT	1	0.971	0.9696	0.884	1	0.733
	TMP vs. TRM	0.260	<0.001	<0.001	<0.001	<0.001	<0.001
	LXT vs. TRM	0.260	<0.001	<0.001	<0.001	<0.001	<0.001

** Shaded cell denotes significant differences between two groups at $P < 0.05$.*

Table V. Independent sample t-test of creep values between 2h & 24h

Time	Material	t-test	*A*	*B*	Y_1	Y_2	*P*	*R*
2h vs. 24h	PT3	t	5.515	16.409	23.015	18.659	20.822	-11.998
		Sig	0.005	<0.001	<0.001	<0.001	<0.001	<0.001
	TMP	t	0.041	6.566	13.849	11.352	12.872	-4.947
		Sig	0.969	0.003	<0.001	<0.001	<0.001	0.008
	LXT	t	6.956	22.610	16.526	19.036	13.757	-21.909
		Sig	0.002	<0.001	<0.001	<0.001	<0.001	<0.001
	TRM	t	-0.397	-3.953	-4.186	-3.998	-2.737	-2.935
		Sig	0.712	0.017	0.014	0.016	0.052	0.043

** Shaded cell denotes significant differences between two groups at $P < 0.05$.*

ferences among LXT, PT3 and TMP ($P>0.05$) except *(A)* stage (Table III).

An immediate decrease in creep strain was seen after removing the load. A time-dependent viscoelastic recovery followed, which approached an equilibrium value of permanent set. The highest recovery *(Y_2)* and permanent set *(P)* were shown by TRM. This was significantly different from the others ($P<0.001$). PT3 showed the highest percentage (65.5%) of recovery *(R)*, followed by TMP (63.4%), LXT (63.4%) and TRM (44.9%), but there were no significant differences among PT3, TMP and LXT ($P>0.05$) (Table IV).

The Second Loading

The kinetic curves of creep strain vs. time were similar to those of first loading. The difference was that the curves shifted to lower on the creep scale except for TRM.

TRM showed the highest initial deformation *(A)*, but not significantly different from the others ($P>0.05$). TRM also showed the highest viscoelastic strain *(B)*, maximum creep strain *(A + B)*, recovery *(Y_2)* and permanent set *(P)* values. This was significantly different from the others ($P<0.001$) (Table IV).

PT3, TMP and LXT showed 0.69% - 0.84% for creep strain *(Y_1)*, 0.56% - 0.71% for recovery *(Y_2)* and 0.13% - 0.16% for permanent set *(P)*. Table IV shows that there were no significant differences among PT3, TMP and LXT in all measuring stages.

The First Loading vs. The Second Loading

Independent samples t-tests showed that PT3, TMP and LXT at the secondary loading showed significantly lower values than those at the first loading for all measuring stages ($P<0.05$), while their percentages of recovery at the secondary loading were higher than those at the first loading ($P<0.05$) (Table V).

However, TRM at the secondary loading showed higher values than at the first loading for all measuring stages. They showed significant differences ($P<0.05$) except at the initial deformation and permanent set stage ($P>0.05$) (Table V).

DISCUSSION

Time-dependent deformation of polymer-based provisional crown and FPD materials is one of the significant clinical aspects in high stress bearing areas, and especially more in continuous stress conditions such as bruxism. Thus, time-dependent creep under constant stress is clinically relevant.[1] This is one of the principal factors that determines the durability of the material.

There are two types of creep. One is *static creep.* This is the time-dependent deformation under constant stress. The other is *dynamic creep.* This is the time-dependent deformation under intermittent, fluctuating stress, like a fatigue test.[2] In the clinical situation, stress is intermittent, and there is the possibility of strain recovery during intervals of stress removal. Although loading patterns are different between that in clinical services and the creep measurements in this study, there is strong correlation between dynamic and static creep behaviour.[3] Thus, continuous compressive stress for static creep behaviour was used in this study.

Applied stress should be lower than that which would cause failure of the material in direct loading. The compressive stress applied in this study was 4 MPa. This stress was much lower than that recommended for amalgam creep testing, 36 MPa (BSI 2938:1985). This was done as the yield stress of the polymer-based provisional crown and FPD materials is lower than that of the amalgam, and higher stress may cause severe deformation or catastrophic failure of the materials without recording a creep strain.

The materials in this study were stored in water

at 37℃, and the testing temperature in this study was 37℃ which represents the human body temperature. The same measurements were carried out on each type of material under the same conditions and with the same equipment.

Viscoelastic behaviour is important to the mechanical performance of polymer-based provisional crown and FPD materials. A creep curve exhibits insight into the elastic, viscous and anelastic response of a viscoelastic material.

All the materials investigated exhibited a time-dependent, viscoelastic creep response, together with substantial anelastic recovery from deformation. The creep curves presented as a strain vs time function showed that the curves levelled out and the creep rate decreased with increasing time. They exhibited plastic deformation (permanent set) after removal of the load. However, this recovery could be complete, provided the conditions of time and surrounding temperature are suitable.[4,5]

Three stages can be seen in creep experiments.[6] An initial rapid deformation stage is followed by a primary creep stage with decelerated creep, and a secondary creep stage with steady creep. A tertiary creep rupture stage can occur due to further deformation. In this study, however, tertiary creep was not shown as the loading time was relatively short.

This study examined two types of polymer-based crown and FPD materials: Three dimethacrylate-based materials (PT3, TMP and LXT) and one monomethacrylate-based material (TRM). A main finding of the time-dependent deformation of these materials was the difference between the monomethacrylate-based and the dimethacrylate-based provisional crown and FPD materials. PT3, TMP and LXT exhibited significantly lower creep strain values in the range of 0.40% - 2.70% and permanent set values in the range of 0.13% - 1.3% than TRM for which strain values were 31.7% - 36.0% and 18.0% - 19.2% respectively ($P<0.05$). According to this result, the null hypothesis that there was no difference in the creep strain between monomethacrylate-based and dimethacrylate-based materials can be rejected.

The differences can be explained in terms of the differences in molecular structure between them. Creep results from the fact that the long polymer chains tend to intermingle. Polymerization reaction of the di-functional monomers give a highly cross-linked polymer which becomes resistant to the movement of polymer chains. Table IV shows that there were no significant differences in creep value, recovery and permanent deformation among PT3, TMP and LXT ($P>0.05$).

In general, all measurements, except the initial deformation stage, significantly decreased with the successive loading ($P<0.05$) in all materials, except TRM which showed an increase in creep deformation and permanent set with successive loading ($P < 0.05$) (Table V). It can be explained by the plasticizing effect of water on TRM. All specimens were tested in water and stored in water. TRM probably absorbed more water than the other materials, and storage in water affects the creep and viscous flow.[7,8]

Applying a load to the polymer-based materials resulted in an instantaneous deformation, followed by a time-dependent deformation or creep. The materials are described as viscoelastic, as they display the properties of both elastic solids and viscous fluids. This property depends upon a variety of factors, such as filler content, filler type, final structure of the organic phase, degree of conversion, diluents, porosity levels, resin volume, resin phase, concentration of cross linking agents, the rate of loading, the environment, the direction of load and the rate of loading, processing condition and temperature.[2,6,9-13]

At a temperature below T_g, the behaviour is predominantly elastic, while above that temperature

it is predominantly viscous or rubber like. In the transition region, both types of response make a significant contribution. The differences in creep rates increase with increasing temperature. These circumstances may be explained by increased thermal motions of the polymer backbones.[13] This test was carried out at 23°C which was below T_g of both materials tested. Thus, all specimen exhibited an elastic deformation.

Composite resins with very hard, rigid fillers, such as quartz, have lower creep than do composite resins with silicate or glass fillers.[12] However, in this test, the effect of fillers on the creep strain of the materials could not be evaluated as its information related to fillers was not available.

Heat-polymerized materials have smaller initial deformation, less creep and quicker recovery than auto-polymerized materials at all temperatures and stress levels, irrespective of composition.[13] It is related to the degree of conversion of the materials. Although all materials tested are auto-polymerized, more creep resistance could be expected when the materials are further polymerized in hot water.[14]

The resistance to creep in a uniaxial compression is superior to the resistance to creep in tension.[13] Less creep resistance can be expected when the specimens are loaded in tension.

The chemical structure appeared to be the most important factor in time-dependent deformation and recovery. This property is influenced by the persistence of the entanglements of the long chain molecule and the ability of the molecules to move past one another.[12] The dimethacrylate-based materials with a highly cross-linked structure showed more creep resistance than the monomethacrylate-based material which allowed movement of the chains with ease under compressive load.

CONCLUSION

(1) The creep experiment was a valuable method for studying the viscoelastic properties of the polymer-based provisional crown and FPD materials.

(2) The dimethacrylate-based provisional crown and FPD materials (PT3, TMP and LXT) showed significantly higher creep resistance and lower deformation than the monomethacrylate-based provisional crown and FPD material (TRM). The ability of the polymer-based provisional crown and FPD materials to resist internal deformation depends upon its chemical component.

(3) In the dimethacrylate-based materials at the secondary loading, creep deformation, recovery and permanent set decreased, and the percentage of recovery increased, while in the monomethacrylate-based material, all values of the measurement increased. This result shows that the secondary loading at 24 hours produced a significant creep magnitude.

(4) TRM showed the highest creep value and deformation (permanent set) which suggests that the material is not suitable for high stress-bearing areas. Thus, monomethacrylate- based materials should not be used in long-term stress-bearing situations.

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