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Viscoelastic stability of pre-cured resin-composite CAD/CAM structures

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ABSTRACT

Objectives. To study the effect of water storage (3 months) on the creep deformation and recovery of CAD/CAM composite materials to determine their viscoelastic stability.

Materials and methods. Five CAD/CAM composite blocks, with increasing filler loading, and one polymer-infiltrated ceramic network (PICN) were studied. Six specimens of each material were separated into two groups (n=3) according to their storage conditions (24 h dry storage at 23°C versus 3 months storage in 37°C distilled water). A constant static compressive stress of 20 MPa was applied on each specimen via a loading pin for 2 h followed by unloading and monitoring strain recovery for a further period of 2 h. The maximum creep-strain (%) and permanent set (%) were recorded. Data were analysed via two-way ANOVA followed by one-way ANOVA and Bonferroni post hoc tests (<0.05) for comparisons between the materials. Homogeneity of variance was calculated via Levene's statistics.

Results. The maximum creep strain after 24 h dry ranged from 0.45% to 1.09% and increased after 3-month storage in distilled water to between 0.71% and 1.85%. The permanent set after 24 h dry storage ranged from 0.033% to 0.15% and increased after 3-month water storage to between 0.087% and 0.18%. The maximum creep strain also reduced with increasing filler loading.

Significance. The PICN material exhibited superior dimensional stability to all of the pre-cured resin composite blocks in both storage conditions with deformation being predominantly elastic rather than viscoelastic. Notwithstanding, two of the resin-matrix composite blocks approached the PICN performance, when dry, but less so after water storage.

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1. Introduction

Resin-based materials designed for use with computer aided design/computer aided manufacturing (CAD/CAM) systems were first introduced as filled or unfilled polymethylmethacrylate (PMMA) with modified polymer networks [1]. New and

improved resin materials have been subsequently developed utilising various matrix and filler constituents [2]. New formulations of CAD/CAM materials aim to provide an enhanced combination of resin-based materials and ceramics utilising their advantageous properties such as durability, strength and colour stability of ceramics; and improved flexural and fatigue properties and low abrasiveness of resins [3–7].

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CAD/CAM resin composites are typically composed of polymer matrices with a high volume fraction of different ceramic fillers such as porcelains, glasses, ceramics, and glass-ceramics. CAD/CAM resin composites are considered as ceramic-like materials in a new classification proposed by Gracis, et al. [8]. However, CAD/CAM composites can be classified based on their micro-structural geometry into two main types: resins with dispersed fillers and polymer infiltrated ceramic networks [PICN] [9].

The main difference between the two is the incorporation method. Simple mixing is used to formulate a composite with dispersed inorganic filler particles, where the matrix resin is formed from photocurable monomers [10]. However, for PICN, a network between ceramic and polymer is created in two steps: first, a porous pre-sintered ceramic network is fabricated and conditioned by a coupling agent. Then this ceramic network is infiltrated with a polymer [11,12]. This manufacturing technique results in a three-dimensional skeleton or a double network hybrid, which enhances the stress distribution and promotes resistance to breakdown of the material [13,14].

Creep can be defined as the strain generated within a material in response to a load application [15]. Depending on the form of this load the creep can be static (constant load application) or dynamic (cyclic load applications) [16]. Static and dynamic creep can be correlated, as reported by some studies [15–18]. The viscoelastic behaviour (creep) of resin composites is influenced by the filler microstructure (volume percent, size and distribution) and the resin matrix composition [15,19]. As a property, it has been studied extensively for dental restorative materials, including amalgam and resin-composites. However, this is the first study of the creep behaviour of CAD/CAM composite blocks.

This study aimed to assess the viscoelastic creep and recovery of different CAD/CAM blocks under compressive loading conditions. The null hypotheses for the investigated materials were that (1) there is no difference in the creep behaviour between the materials (2) there is no effect of water storage (3 months) on the creep deformation of CAD/CAM composite materials and (3) the creep behaviour will not be affected by their composition (filler weight percentage).

2. Materials and methods

Five CAD/CAM composite blocks and one polymer infiltrated ceramic network (PICN) were investigated. The details of manufacturers' compositional information and experimentally determined filler weight percentages are given in Table 1. In total 36 cylindrical specimens were prepared. CAD/CAM blocks were sectioned and shaped into cylinders of 4 mm diameter and 6 mm height using a diamond blade (MK 303, MK diamond, CA, USA) mounted on a saw (Isomet 1000 Precision Cutter; Buehler Co, IL, USA) under constant water irrigation. All specimens were wet-ground and polished with silicon carbide papers (SiC) P500, P1200 (Buehler Co, Illinois, USA). For each material, the specimens were divided into two groups (three specimens in each group, $n=3$) for storage, as follows: Group one: 24 h dry at 23 °C; Group two: 3 months in 37 ± 1 °C distilled water.

Table 1 – Materials investigated manufacturer's compositional information and experimentally determined filler weight percentage.

Materials (code)	Manufacturer	Composition by weight represented by manufacturers polymer filler	Filler composition by weight (%) determined by ashing (SD) [36]
Polymer infiltrated ceramic network (PICN)	VitaEnamic (EN)	14% UDMA + TEGDMA	85.1 (0.1)
Resin composite CAD CAM blocks	Grandio Blocs (GR)	14% UDMA + DMA	84.5 (0.01)
	Lava™-Ultimate (LU)	20% (Bis-GMA, UDMA, Bis-EMA, TEGDMA)	74.8 (0.1)
	BRILLIANT-Crios (BC)	Cross-linked methacrylates (Bis-GMA, Bis-EMA, TEGDMA)	70.1 (0.05)
Cerasmart (CS)	GC dental products, Europe	Bis-MEPP, UDMA, DMA	66.1 (0.2)
Block HC (HC)	Shofu	UDMA + TEGDMA	63 (0.02)

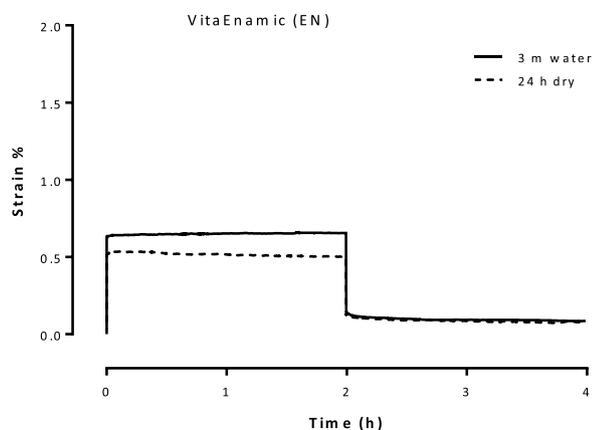


Fig. 1 – Creep and recovery monitored in real time after 24 h dry and three months water storage of Enamic (EN).

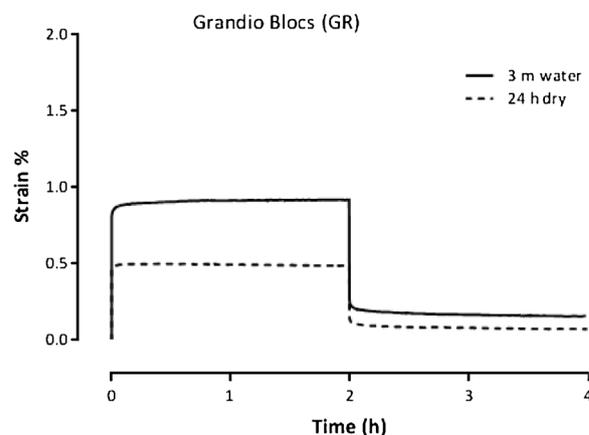


Fig. 2 – Creep and recovery monitored in real time after 24 h dry and three months water storage of Grandio blocs (GR).

The static creep deformation of the CAD/CAM composite blocks was measured using a creep apparatus as described previously [20]. A constant compressive stress of 20 MPa was applied on each specimen via a loading pin for 2 h followed by load removal for a further measurement period of 2 h (total: 4 h). The creep strain and recovery were monitored continuously in real time using a LVDT transducer system [16,20]. The maximum creep-strain (%), and permanent set (%) were obtained after loading and recovery respectively. Maximum creep strain, maximum creep recovery, percentage creep recovery and permanent set were obtained from the creep/time plots.

Data were analysed using statistical software (SPSS ver. 23, SPSS Inc., Illinois, USA). Homogeneity of variance was calculated by Levene's statistics. Two-way ANOVA (2 factors; material and storage) followed by one-way ANOVA and the Bonferroni post hoc tests were used for comparisons between the materials for each group. Independent sample t-test was used for the difference between the two storage groups for each individual material. All tests were conducted at a significance level of $\alpha = 0.05$.

3. Results

The results are presented in Figs. 1–11 and Table 2. Figs. 1–6 show the creep and recovery in real time after 24 h of dry storage and after three months of water storage for the six CAD/CAM blocks.

There was a statistically significant difference in creep behaviour between the investigated materials. The maximum creep strain after 24 h storage ranged from 0.45% to 1.09% and increased after 3 m storage in distilled water to between 0.71% and 1.85%. The permanent set after 24 h storage ranged from 0.033% to 0.15% and increased after 3 m storage in distilled water to between 0.087% and 0.18%. The percentage creep recovery after 24 h storage ranged from 85.01% to 96.0% and slightly reduced after 3 m storage in distilled water to between 83.5% and 94.05%. Figs. 7–10, show maximum creep strain, permanent set, maximum creep recovery, and percentage creep recovery of the two groups (24 h dry and 3 m water storage) of the CAD/CAM composite blocks. Increased filler loading

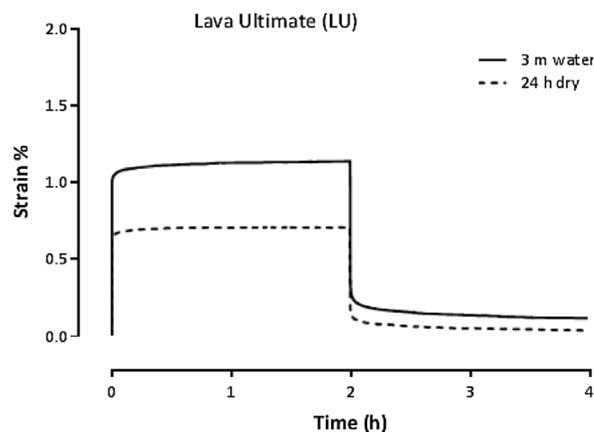


Fig. 3 – Creep and recovery monitored in real time after 24 h dry and three months water storage of LavaUltimate (LU).

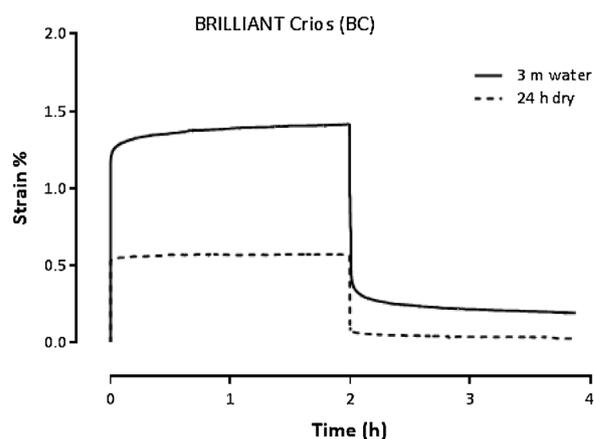


Fig. 4 – Creep and recovery monitored in real time after 24 h dry and three months water storage of BRILLIANT Crios (BC).

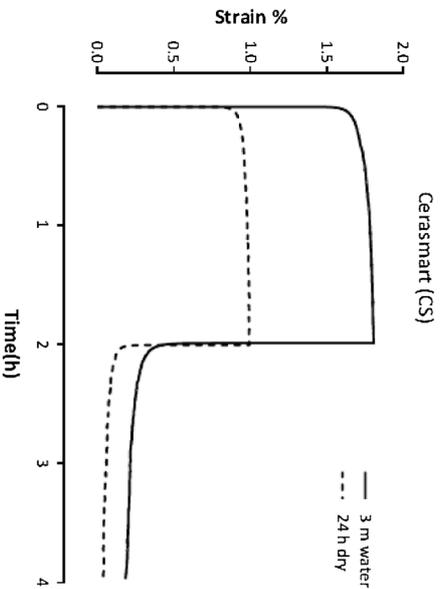


Fig. 5 – Creep and recovery monitored in real time after 24 h dry storage and three months water storage of Cerasmart (CS).

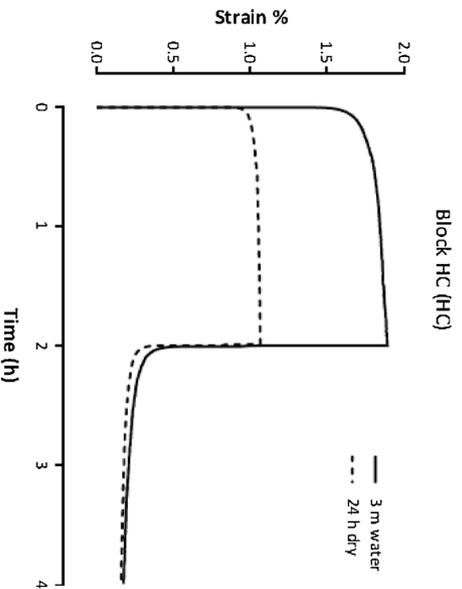


Fig. 6 – Creep and recovery monitored in real time after 24 h dry and three months water storage of Block HC (HC).

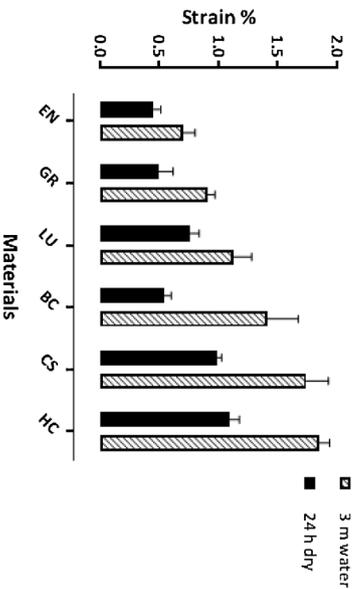


Fig. 7 – Maximum creep strain (%) of the two groups (24 h dry and 3 m water storage) of CAD/CAM composite blocks.

Table 2 – Maximum creep strain (%), permanent set (%), maximum creep recovery (%), and maximum recovery percentage after 24 h dry and three months water storage.

Storage		24 h dry storage at 23 °C				Three month water storage at 37 °C			
Material		Max. creep strain(%)	Permanent set (%)	Max. creep recovery (%)	Percentage of creep recovery	Max. creep strain (%)	Permanent set (%)	Max. creep recovery (%)	Percentage of creep recovery
PICN	EN	0.447 ^{a,A} (0.07)	0.065 ^{a,A} (0.01)	0.38 ^{a,A} (0.06)	85.01 ^{a,A} (2.1)	0.71 ^{a,B} (0.1)	0.087 ^{a,A} (0.07)	0.62 ^{a,B} (0.04)	87.3 ^{a,A} (8.2)
	GR	0.50 ^{a,b,A} (0.12)	0.065 ^{a,A} (0.03)	0.43 ^{a,b,A} (0.09)	86.0 ^{a,A} (4.30)	0.91 ^{a,B} (0.06)	0.15 ^{a,A} (0.08)	0.76 ^{a,d,B} (0.13)	83.5 ^{a,A} (10.7)
Resin composite	LU	0.763 ^{b,d,A} (0.08)	0.054 ^{a,A} (0.03)	0.654 ^{b,d,A} (0.11)	86.05 ^{a,A} (6.2)	1.13 ^{a,c,B} (0.15)	0.12 ^{a,A} (0.07)	1.02 ^{c,B} (0.17)	90.2 ^{a,A} (7.19)
	BC	0.55 ^{a,d,A} (0.06)	0.044 ^{a,A} (0.01)	0.50 ^{a,d,A} (0.06)	90.9 ^{a,A} (3.90)	1.42 ^{b,c,B} (0.26)	0.18 ^{a,A} (0.1)	1.22 ^{b,c,d,B} (0.30)	85.9 ^{a,A} (11)
CAD CAM blocks	CS	0.99 ^{c,A} (0.04)	0.033 ^{a,A} (0.01)	0.95 ^{c,A} (0.03)	96.0 ^{a,A} (0.93)	1.74 ^{b,B} (0.19)	0.14 ^{a,A} (0.09)	1.56 ^{b,B} (0.10)	89.7 ^{a,A} (5.3)
	HC	1.09 ^{c,A} (0.09)	0.15 ^{a,A} (0.01)	0.91 ^{c,A} (0.09)	83.5 ^{a,A} (9.80)	1.85 ^{b,B} (0.09)	0.17 ^{a,A} (0.04)	1.74 ^{b,B} (0.06)	94.05 ^{a,A} (1.9)

VitaEnamic (EN); Grandio Blocs (GR); Lava™-Ultimate (LU); BRILLIANT Crios (BC); Cerasmart (CS); BlocK HC (HC). Values with the same small superscript letters represent non-significant difference among different materials (Bonferroni post hoc tests ($\alpha=0.05$)). Values with the same capital superscript letters represent non-significant difference among different behaviour for each material, independent sample t test. ONEWAY significance: max. creep strain(%) at (24h): $p < 0.05$; permanent set (%): $p = 0.1$; max. creep recovery (%): $p < 0.05$; percent creep recovery: $p = 0.1$. *Max. creep strain (%) at (3 m): $p < 0.05$; permanent set (%): $p = 0.7$; max. creep recovery (%): $p < 0.05$; percent creep recovery: $p = 0.7$.

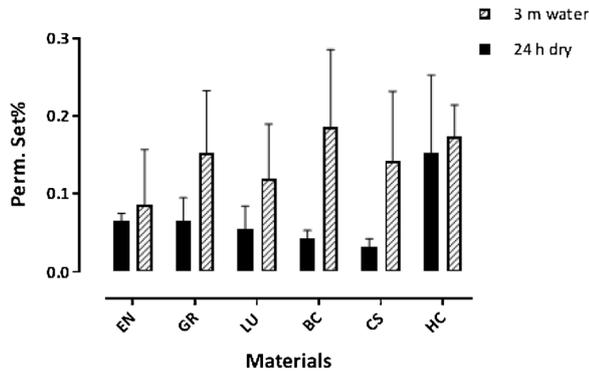


Fig. 8 – Permanent set (%) of the two groups (24 h dry and 3 m water storage) of CAD/CAM composite blocks.

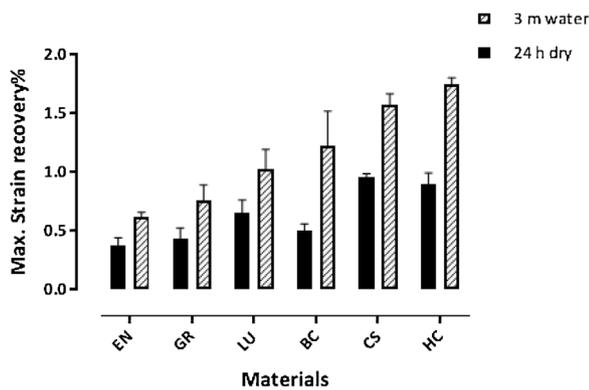


Fig. 9 – Maximum creep recovery (%) of the two groups (24 h dry and 3 m water storage) of CAD/CAM composite blocks.

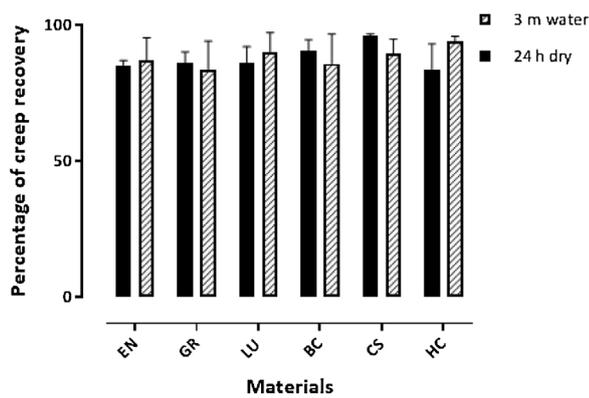


Fig. 10 – Percentage of creep recovery of the two groups (24 h dry and 3 m water storage) of CAD/CAM composite blocks.

decreased the creep strain. Fig. 11, shows a negative correlation and linear regression between maximum strain and experimentally-determined filler weight percentage after 24 h dry storage $R^2 = 0.74$, $p = 0.02$ and 3 m water storage $R^2 = 0.96$, $p = 0.0007$.

A two-way ANOVA was conducted that examined the effect of storage and material type. There was a statistically significant interaction between the effects of storage and material,

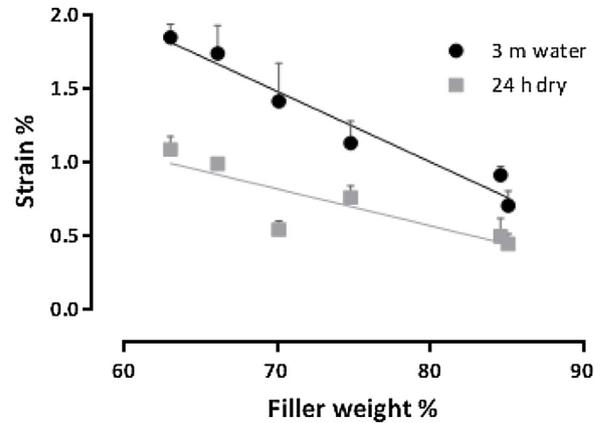


Fig. 11 – A scatter plot showing a negative correlation and linear regression between filler weight percentage (the filler weight percentage were as determined experimentally, rather than the manufacturer's figures) and maximum strain percentage after 24 h dry storage $R^2 = 0.74$, $p = 0.02$ and 3 m water storage $R^2 = 0.96$, $p = 0.0007$.

$F(5,24) = 6.178$, $p = 0.0008$. A significant effect of storage and material type ($p < 0.0001$) was found. Simple main effects analysis showed that material effect was significantly more in 3 m water storage than the material effect in 24 h dry storage ($p \leq 0.03$).

4. Discussion

CAD/CAM resin composites have some improved mechanical properties [21] compared to conventional indirect composites. They have better wear resistance [22,23], flexural strength [24], fracture toughness, and fracture strength [3,11]. This can be attributed to the higher degrees of conversion of CAD/CAM composites [25,26] as they are polymerised under high pressure and high temperature [27,28]. This results in higher composite homogeneity and reliability with fewer flaws and pores compared to conventional indirect composites [27,29] that enables incorporation of higher filler content [30]. Further, when compared to all-ceramic materials, CAD/CAM composites are less hard and less stiff, so the opposing enamel exhibits less wear clinically. Also, they are easily fabricated and repaired [31].

This study investigated five CAD/CAM composite blocks and one polymer infiltrated ceramic network (PICN) with different filler loadings and resin matrices. There was a statistically significant difference in the creep/recovery behaviour between the investigated materials. Thus the first null hypothesis was rejected.

In this study, the specimens of Group two; stored for 3 months in $37 \pm 1^\circ\text{C}$ distilled water in the incubator, exhibited higher creep strain and recovery than those stored dry for 24 h at 23°C . A significant effect of water storage and material type ($p < 0.0001$) was found. Therefore the second null hypothesis was rejected. Storage in water can lead to a reduction in material stiffness due to plasticization of the polymer matrix [32,33]. Water sorption increased creep and reduced

creep recovery of the composite materials. This corresponds to some previous studies [17,34,35].

For the purpose of correlation, plots with filler loading (w/w), from previously published data on filler content were utilized [36]. There was a negative correlation between maximum creep percentage and filler weight percentage. However, this correlation was more statistically significant for Group two: water storage for 3 m ($R^2 = 0.96$, $p = 0.0007$) compared to the Group one: dry storage for 24 h ($R^2 = 0.74$, $p = 0.02$). Therefore the third null hypothesis was rejected.

The PICN material (EN) showed higher creep resistance, attributable to the robust ceramic–matrix microstructural geometry of PICN as compared to other CAD/CAM composite blocks with resin incorporating dispersed ceramic fillers. However, both EN, and GR had comparable creep resistance and permanent set under both dry and wet conditions. HC with the lowest filler content (63% w/w) showed the highest creep strain followed by CS (66% w/w) and BC (70% w/w). LU (75% w/w) had a middle-ranking creep strain.

These findings are in agreement with a Hertzian indentation study of two experimental PICN materials with different filler loadings compared to ceramics. The PICN with lower filler incorporation had greater indentations. Both PICN materials had greater indentations than that of investigated ceramics [37].

All investigated materials exhibited minimal permanent set and high recovery. Both characteristics were not significantly different between the investigated materials, reflecting the material resistance to any permanent change. The maximum permanent set obtained after 3 months of water storage was 0.18 % by BC, which has (70% w/w) but it was not significantly different from all other investigated materials.

The resistance to creep and the recovery were generally higher than that of conventional composites. They also exhibited a lower permanent set. A recent study on bulk fill composites with immediate measurement after immersion in different media using 3-point bending, showed higher permanent set especially in water up to 1.14%, and lower recovery ranging from 45 to 64% [38]. Many studies showed that conventional composites, particularly some bulk fill types with as high a filler loading as our investigated materials, exhibit higher creep strain even in shorter-term wet storage [18,35,38].

In this study, the deformation was predominantly elastic rather than viscoelastic as expected in conventional composites attributable to the higher degree of conversion and improved mechanical properties of CAD/CAM composite blocks.

Maximum creep recovery depends upon the level of maximum creep strain; hence both properties have a similar trend in terms of highest to lowest values. Creep can range from 1–6% depending on the filler content [20,39]. All investigated materials showed less than 1% creep under dry conditions and less than 2% after wet storage for 3 months. Compressive creep resistance reflects the materials viscoelastic stability and its resistance to catastrophic failure under loading [16,40]. The applied stress in this study (20 MPa) was similar to the average *in-vivo* bite force [41] and corresponds with the maximum occlusal force intraorally during occlusion [42].

5. Conclusions

- The PICN material exhibited superior dimensional stability to all of the precured resin composite blocks with deformation being predominantly elastic rather than viscoelastic.
- Creep deformation and maximum recovery demonstrated lower viscoelastic stability of pre-cured CAD/CAM composite blocks upon water storage.
- Pre-cured CAD/CAM composite showed better viscoelastic stability compared to conventional direct or indirect resin-composites.

Data availability

The data used to support the findings of this study are included within the article.

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