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FLEXURAL STRENGTH OF PROVISIONAL RESTORATIVE MATERIALS UPON AGING

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Background: Aging may affect strength of provisional restorative materials.
Objective: This study evaluated the effect of aging on strength of heat-polymerized polymethyl methacrylate (Hp-PMMA), auto-polymerized (Ap) PMMA, bis-phenyl-glycidyl dimethacrylate (Bis-GMA), and computer-aided design/computer-aided manufacturing (CAD/CAM) containing either PMMA or acrylate resin.
Methods: Two hundred-ten bars (2x2x25mm) were fabricated from Hp-PMMA: Major C&B (M); Ap-PMMA: Unifast™ (U); Bis-acryl: Protemp™ (P), Luxatemp® (L); PMMA-CAD/CAM: Telio® CAD (T), artBloc® (R); and acrylate-CAD/CAM: Vita CAD Temp® (V). Each was divided into aging- (A) and non-aging- (N) groups (n=15 each). A-groups were thermo-cycled (5°C v.s 55°C, 30 sec each, 5000 cycles). Flexural strength was determined in universal testing machine at 1 mm/min crosshead speed, 50N/min loading. An analysis of variance (ANOVA) and Bonferroni's test was determined for significant difference (α=0.05). Weibull statistics were determined for Weibull modulus (m), and characteristics strength (σ_o). Scanning electron micrographs (SEM) were examined for fracture surfaces.
Results: The values (means±sd (MPa), m, σ_o) were (84.62± 3.73, 25.23, 86.53) and (84.05± 6.39, 13.21, 87.28) for V_N and V_A, (133.49± 4.32, 34.09, 135.54) and (123.11± 4.55, 28.76, 125.35) for T_N and T_A, (120.59± 6.94, 19.01, 123.84) and (119.96± 6.90, 19.21, 123.16) for R_N and R_A, (94.35± 4.07, 25.82, 96.24) and (93.07± 3.22, 32.19, 94.58) for P_N and P_A, (110.60± 6.20, 19.99, 113.44) and (97.23± 7.77, 13.82, 100.78) for L_N and L_A, (114.30± 5.21, 23.90, 116.79) and (112.21± 5.70, 19.86, 115.13) for MN and MA, and (89.45± 2.96, 32.77, 90.88) and (84.96± 5.33, 17.66, 87.42) for U_N and U_A respectively. T revealed the highest, whereas V possessed the lowest strength for both N- and A- condition. Aging significantly affected strength.
Conclusions: Flexural strengths were differences among materials. PMMA-CAD/CAM possessed the highest, while acrylate-CAD/CAM possessed the lowest. Hp-PMMA showed better strength than Ap-PMMA. Bis-acryl resin was stronger than Ap-PMMA. Aging reduced strength for all materials tested.

***Keywords*:** CAD/CAM, flexural strength, provisional restorative materials, polymer, resin

1. INTRODUCTION

Provisional restorations have been serving as the blueprint for fixed prostheses rehabilitations, which extremely require ultimate strength to be in function during the treatment period (Santosa 2007). They are capable of preventing tooth movement, protecting pulp vitality, providing stable occlusion, and rendering aesthetics. They allow practitioner for determination vertical dimension, phonetics, appearance and patient satisfaction (Rayyan et al. 2015, Clenaland et al. 2012, Almeida et al. 2016, Blank 2012). The provisional material that possesses superior strength are preferred for long-span bridges (Almeida et al. 2016). Un-durable interim restoration can fracture easily as it subjected to masticatory force (Hyde et al. 2007). The breakage of restoration at the connector and cervical area were reported (Patras et al. 2012). Although, it can be repaired, it is considerable time-consumed process (Burns et al. 2003). Provisional restorative materials can be categorized upon different chemical structures as conventional acrylic resins, bisphenyl acrylate (Bis-acryl)

resin, visible light-polymerized resin, and miscellaneous (Clenaland et al. 2012, Haselton et al. 2005). The provisional resin acrylic either auto-polymerization (Ap) or heat-polymerization (Hp) that comprises of methacrylate connected with various esters of poly-acids can generates various forms of methacrylate such as poly-methyl methacrylate (PMMA) and ethyl methacrylate (EMA) (Anusavice et al. 2013). The PMMA is a non-crystalline structure, comprising methyl ester and carboxyl group that provides color stability and marginal adaptation. The EMA that has shorter polymer chain, tends to possess lesser strength than MMA (Anusavice et al. 2013, Christensen 2004, Astudillo-Rubio et al. 2018). The Bis-acryl resin, primarily comprised with bis-phenyl-glycidyl dimethacrylate (Bis–GMA) forming densely cross-linked structures, is considerably easy handling, in auto- or dual-polymerization, color stable, and minimal shrinkage (Rayyan et al. 2015, Clenaland et al. 2012). The light-polymerized resin, is mainly composed of urethane di-methacrylate (UDMA), illustrates remarkable strength, but compromised marginal fit. Others; such as polycarbonate crown form are seldom used.

Provisional restorations are customarily fabricated by mixture of powder and liquid or base and catalyst. Recently, the digitally fabricated provisional restoration from computer aided design/computer aided manufacturing (CAD/CAM) has been developed by using CAD/CAM block containing PMMA or acrylate polymer (Ender et al. 2016, Stawarczyk et al. 2016). The digitally fabricated- provides better result than conventionally fabricated- provisional restoration in term of color stability, wear resistance, and homogeneity of material (Stawarczyk et al. 2016). Selection material usually based on material compositions, color stability, and fabrication process (Rayyan et al. 2015, Burns et al. 2003, Haselton et al. 2005, Vichi et al. 2004). Provisional resin is prone to absorb water leading to weakness of restoration due to the degradation of covalent bond in polymeric chains (Burns et al. 2003, Haselton et al. 2005, Vichi et al. 2004).

Aging is an auto-degradation process upon certain period of time, which related to the chemical and mechanical properties of materials. Long-term use of materials is related with the performance threshold of material to endure stress during function in oral environments (Yao et al. 2014, Alt et al. 2011, Almeida et al. 2016, Kadiyala et al. 2016). Flexural strength of provisional restorative materials were still controversies (Yanikoğlu et al. 2014, Gujjari et al. 2013, Rayyan et al. 2015, Poonacha et al. 2013). It was reported that Ap-PMMA is stronger than Bis-acryl (Poonacha et al. 2013). Others reported that Bis-acryl is stronger than Ap-PMMA (Yanikoğlu et al. 2014, Hamza et al. 2014, Alt et al. 2011, Gujjari et al. 2013). The PMMA-CAD/CAM block was reported of the highest flexural strength, followed by Bis-acryl and conventional PMMA respectively (Rayyan et al. 2015, Gülce et al. 2017). Differences of materials, and aging may influence on strength of provisional materials. This study compared flexural strength of different provisional materials: Ap-PMMA, Hp-PMMA, Ap-Bis-acryl, PMMA-CAD/CAM and acrylate-CAD/CAM upon aging. It was hypothesized of no significantly differences in flexural strength of different provisional materials upon aging.

2. MATERIALS AND METHODS

Seven provisional materials were fabricated from Hp-PMMA: Major C&B (M, Major Prodotti Dentari SPA, Moncalieri,Italy); Ap-PMMA: Unifast™ Trad (U, GC Corp., Tokyo, Japan); Bis-acryl: Protemp™ IV (P, 3M ESPE, MN, USA), Luxatemp® star (L, Zenith-DMG/ Foremost Dental, NJ, USA); PMMA-CAD/CAM: Telio® CAD (T, Ivoclar Vivadent, Bensheim, Germany), artBloc® (R, Merz Dental GmbH, Lutienburg, Germany); and acrylate-CAD/CAM: Vita CAD Temp® (V, Vita Zahnfabrik, Bad Säckingen, Germany) in bar-shaped of 25x2x2 mm (ISO 10477:2004) (ISO10477:2004) The CAD/CAM blocks of V and R were cut into specific sizes by Isomet®1000 (Buehler Ltd., Lake Bluff, IL, USA). The Bis-acryl specimens for P and L groups were prepared from injectable materials in a stainless steel mold. The Hp-PMMA specimens for M groups were prepared from mixing resin polymer to liquid monomer at ratio 1:2, packed in a stone mold, and heat polymerized at 70°C for 8 hours. The Ap-PMMA specimens for U groups

were prepared from mixing resin polymer to liquid monomer at ratio1:2, packed in a stainless steel mold, at 2.0-2.5 psi. All samples were polished with silicon carbide abrasive in the polishing machine (Ecomet™3, Buehler Ltd., Lake Bluff, IL, USA). Samples of each group were divided in to aged (A-) and non-aged(N-) groups. Samples in A-group were prepared by immersion in artificial saliva (1 L double-distilled H2O, 1.6802 g NaHCO3, 0.41397 g NaH2PO4·H2O, and 0.11099 g CaCl2) for 24 hours (Poonacha et al. 2013, Haselton et al. 2005, Sodata et al. 2017) , and thermocycling at 5°C and 55°C for 5000 cycles, 30 sec dwelling time prior to flexural strength test.

2.1 Determination of flexural strength

The specimens were subjected to evaluate flexural strength using three-point bending test, having two supporting bars laid 20 mm (L) apart with one vertically movable rod at the center of the supporting bars, in the universal testing machine (Instron ElectroPuls E1000, Instron Corp., Norwood, MA, USA) at a crosshead speed of 1 mm/min with 50N/min loading. Load at failure was recorded and calculated for flexural strength using equation (1), in which σ: Flexural strength (MPa), F: Load (N), L: Supporting length (mm), b and h: Width and height of specimen (mm).

$$\sigma = 3FL/2bh^2 \qquad (1)$$

2.2 Scanning Electron Photomicrograph

Fractured surface of samples were ultrasonically cleaned in distilled water, dried in the desiccator (Northman, Taipei Hsein, Taiwan), gold-palladium coated in sputter coater (K500X Emitech, Ashford, England) at a current of 10 mA and vacuum 130 mTorr for 3 minutes, and evaluated in the scanning electron microscope (SEM, Hitachi S-3000N, Osaka, Japan) at 500xmagnification.

2.3 Statistical analysis

The data were analyzed using SPSS/PC Version 22 (IBM, Armonk, NY, USA). An analysis of variance (ANOVA) was applied to determine the significant differences in flexural strength. Bonferroni's multiple comparisons were used to determine for difference among groups (α=0.05). The weibull analysis was performed for reliability of strength using Weibull®statistics (ReliaSoft, Tucson, AZ, USA) to estimate for Weibull modulus (m), and characteristic strength (σ_o).

3. RESULTS

The means, standard deviations (sd), 95% confidence interval (CI), Weibull modulus (m), and characteristic strength (σ_o) for each material were presented in Table 1, Figure 1(A, B). Flexural strength of all materials decreased upon aging. The highest flexural strength (MPa) of N- group was indicated with T_N (133.49± 4.32), followed by R_N (120.59±6.94), M_N (114.30±5.21), L_N (110.60±6.20), P_N (94.35±4.07), U_N (89.45±2.96), and V_N (84.62±3.73), whereas the highest flexural strength of A-groups was demonstrated with T_A (123.11±4.55), followed by R_A (119.96±6.90), M_A (112.21±5.70), L_A (97.23±7.77), P_A (93.07±3.22), U_A (84.96±5.33), and V_A (84.05±6.39). T group possessed the highest strength, vice versa for V groups for both N- and A-condition. ANOVA indicated significantly affected flexural strength upon different materials and aging (P<0.05), and interaction of two factors (P<0.05) as shown in Table 2, Figure 2(A, B). Bonferroni's multiple comparisons indicated significant differences in flexural strengths of various materials (P<0.05), except for V and U group (P>0.05) as shown in Table 3. Aging significantly reduced flexural strength of all provisional material tested (Figure 2B). There was no significant difference between T_A and both R_N, and R_A as well as among the groups of U_N, V_A, V_N, P_A, and P_N (P>0.05). The U_A significantly reduced in flexural strength than P_A, P_N, L_A, and L_N (p<0.05), but no significant difference between V_A and V_N ($P>0.05$). No significant differences

among groups of R_N, R_A, M_N, M_A, and L_N were indicated. The R and M exhibited significantly high reduction in flexural strength after aging ($P<0.05$), while L_A revealed no significant difference from P_A and P_N ($P>0.05$) as shown in Table 4.

The SEM of the N-fracture surfaces (Figure 3 (A-G)) and the A-fracture surfaces (Figure 3 (H-N)) exhibited differences in mixed mode fracture surface characteristics as a result of different materials. The V_N, V_A showed distinguished micro-filler reinforced polyacrylic, surrounded by acrylate polymer. Small grains in irregular surface were noted. The V_A was less density than V_N. The T_N, and T_A exhibited discrete roughness accommodated with smooth and dense matrix. Fracture surface of T_N was consistent-line pattern, while T_A exhibited mixed pattern. The R_N, and R_A exhibited similar fracture pattern as T_A group with small grain embedding in the matrix. The P_N, P_A exhibited both irregular and smooth surface. The crack line was relatively straight with slight deviations. The L_N, and L_A exhibited irregular surface roughness. The M_N exhibited sharp crack line with surface roughness.

Table 1: Mean, standard deviation(SD), 95% confidential interval (CI), Weibull modulus(m), characteristic strength (σ_o) of provisional materials upon aging (A) and non-aging (N).

Group	Material	Treatment	N	Flexural strength				m	(σ_o)
				Mean&SD		95% CI			
				Mean	SD	Lower	Upper		
V_N	Vita CAD Temp®	N	15	84.62	3.73	82.55	86.68	25.32	86.35
V_A	Vita CAD Temp®	A	15	84.05	6.39	80.51	87.59	13.21	87.28
T_N	Telio® CAD	N	15	133.49	4.32	131.10	135.88	34.09	135.54
T_A	Telio® CAD	A	15	123.11	4.55	120.58	125.63	28.76	125.35
R_N	artBloc®	N	15	120.59	6.94	116.75	124.43	19.01	123.84
R_A	artBloc®	A	15	119.96	6.90	116.14	123.78	19.21	123.16
P_N	Protemp™ IV	N	15	94.35	4.07	92.09	96.60	25.82	96.24
P_A	Protemp™ IV	A	15	93.07	3.22	91.29	94.85	32.19	94.58
L_N	Luxatemp® star	N	15	110.60	6.20	107.16	114.03	19.99	113.44
L_A	Luxatemp® star	A	15	97.23	7.77	92.93	101.53	13.82	100.78
M_N	Major C&B-V	N	15	114.30	5.21	111.42	117.19	23.90	116.79
M_A	Major C&B-V	A	15	112.21	5.70	109.05	115.36	19.86	115.13
U_N	Unifast™ Trad	N	15	89.45	2.96	87.81	91.09	32.77	90.88
U_A	Unifast™ Trad	A	15	84.96	5.33	82.01	87.91	17.66	87.42

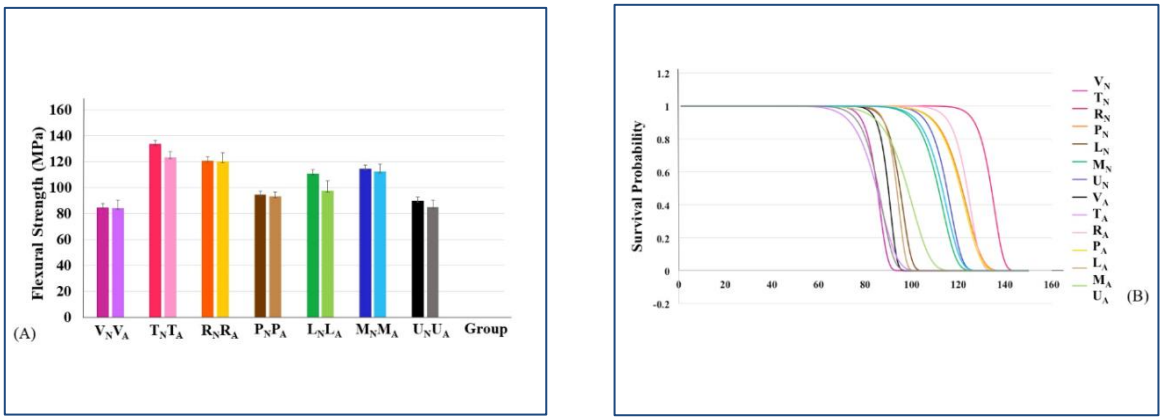

Figure 1: Flexural strength (A) and Weibull curves (B) of provisional materials upon aging.

Table 2: ANOVA of flexural strength of provisional materials upon aging

Source	SS	Df	MS	F	P Value
Corrected Model	53782.686[a]	13	4137.130	140.434	.000
Intercept	2290040.836	1	2290040.836	77735.032	.000
Materials	51431.668	6	8571.945	290.973	.000
Aging	1153.470	1	1153.470	39.154	.000
Materials * Aging	1197.548	6	199.591	6.775	.000
Error	5774.076	196	29.460		
Total	2349597.599	210			

Table 3: Multiple comparisons of strength of different provisional materials

Group	V	T	R	P	L	M	U
V	1	.00	.00	.00	.00	.00	.87
T		1	.00	.00	.00	.00	.00
R			1	.00	.00	.00	.00
P				1	.00	.00	.00
L					1	.00	.00
M						1	.00
U							1

Table 4: Multiple comparisons of strength of different provisional materials upon aging

Group	**V_N**	**T_N**	**R_N**	**P_N**	**L_N**	**M_N**	**U_N**	**V_A**	**T_A**	**R_A**	**P_A**	**L_A**	**M_A**	**U_A**
V_N	1	.00	.00	.00	.00	.00	1.00	1.00	.00	.00	.00	.00	.00	1.00
T_N		1	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00	.00
R_N			1	.00	.00	.16	.00	.00	1.00	1.00	.00	.00	.00	.00
P_N				1	.00	.00	1.00	.00	.00	.00	1.00	1.00	.00	.00
L_N					1	1.00	0.00	0.00	0.00	0.00	0.00	0.00	1.00	0.00
M_N						1	.00	.00	.00	.44	.00	.00	1.00	.00
U_N							1	.64	.00	.00	1.00	.01	.00	1.00
V_A								1	.00	.00	.00	.00	.00	1.00
T_A									1	1.00	0.00	0.00	0.00	0.00
R_A										1	.00	.00	.01	.00
P_A											1	1.00	0.00	0.01
L_A												1	.00	.00
M_A													1	.00
U_A														1

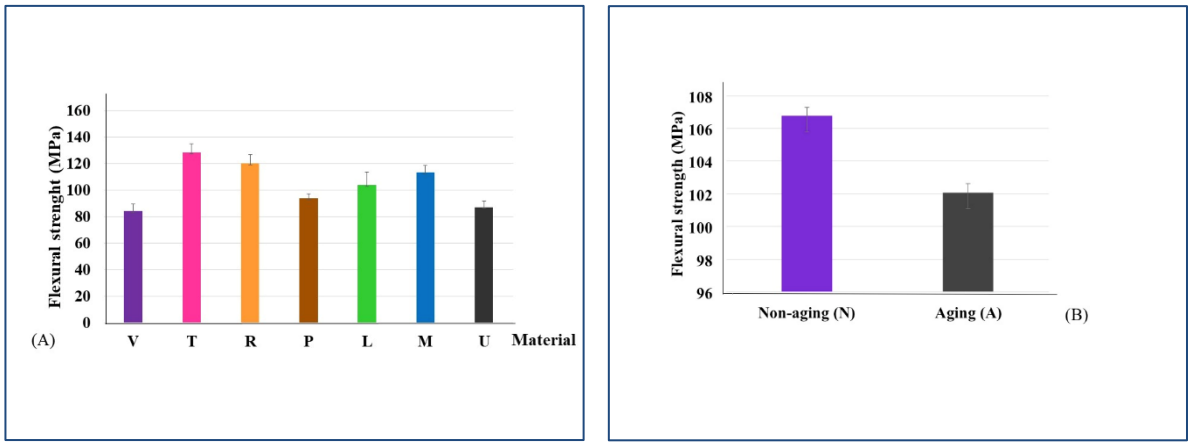

Figure 2: Flexural strength of different provisional materials (A) upon aging (B).

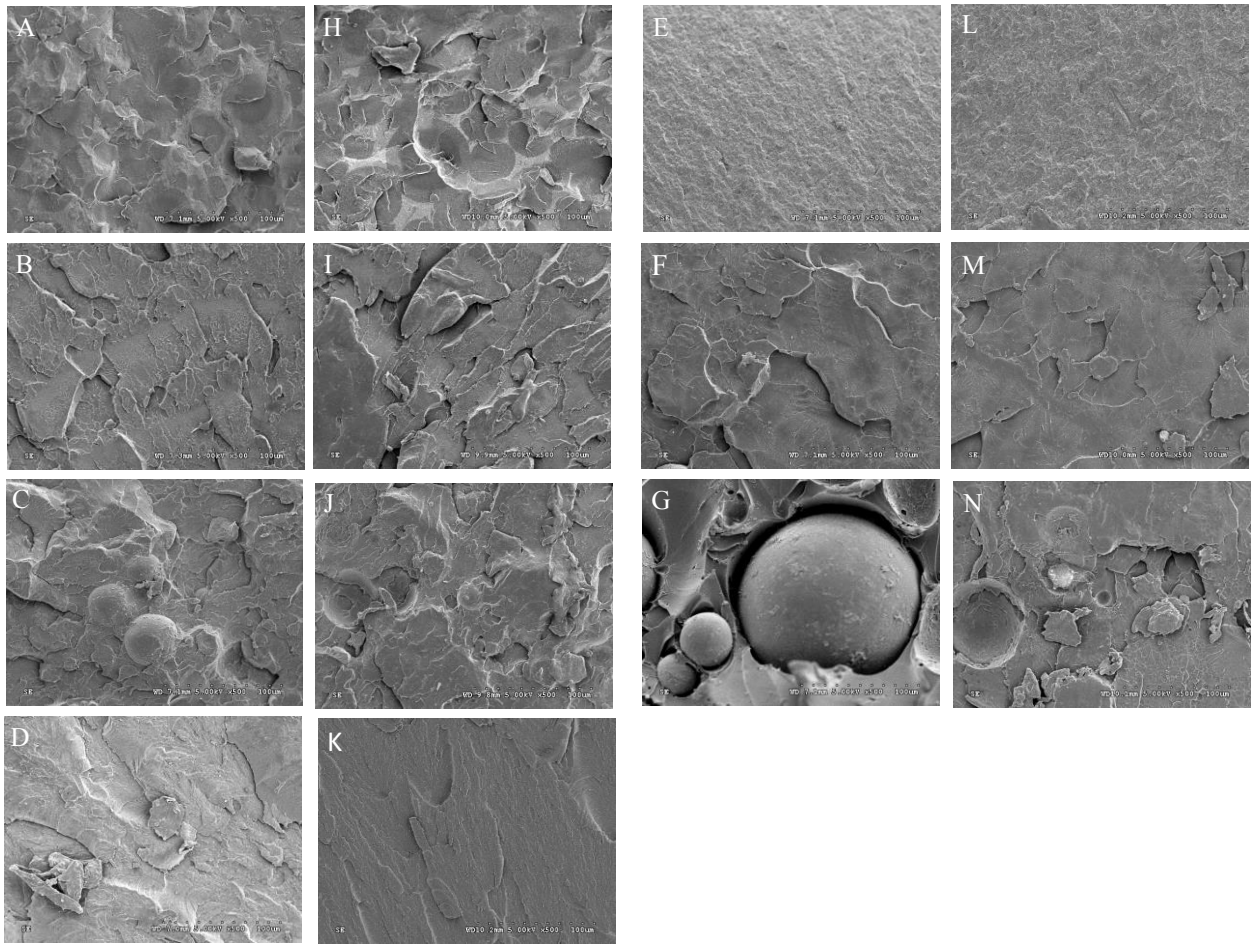

Figure 3: SEM of non-aged (A, B, C, D, E, F, G) and aged (H, I, J, K, L, M, N) of Vita CAD Temp® (A, H), Telio® CAD (B, I), artBloc®(C, J), Protemp™ IV (D, K), Luxatemp® (E, L), Major C&B (F, M), Unifast™ Trad (G, N) at 500x

4. DISCUSSION

This study manifested that types of provisional material and aging influenced flexural strength. Thus, the null hypothesis was rejected. Decreasing in flexural strength upon aging was evidenced. The differences in flexural strength were attributed to types of materials, duration of

material in service, material manipulation and residual monomer, and defect (Kadiyala et al. 2016). The study exhibited that Bis-acryl resin has higher flexural strength than Ap-PMMA, but lower than Hp-PMMA. This may be related to differences in material composition and the method of manipulation. The MMA-based resin is mono-function and linear molecules that seem to uptake water into the polymer network. Whereas the structure of Bis-acryl resin is a cross-linked polymeric chain, comprising organic matrix, inorganic filler particles, and multifunctional monomers (Bis-GMA and TEGDMA). This possibly provides the durable structure to withstand breaking- and aging- stress for Bis-acyl resin (Kadiyala et al. 2016).

The difficulty in controlling void and porosity upon fabrication process Ap-PMMA leads to lower flexural strength than Bis-acryl resin, Hp-PMMA and PMMA-CAD/CAM block. Hp-PMMA was capable of elimination excess residual monomer approximately 0.5%, resulted in higher degree of polymerization and rendering stronger material (Kadiyala et al. 2016). The PMMA-CAD/CAM blocks are industrialized product that possessed less free monomer, less porosity, and more homogeneous structure, thus the strength was higher than others (Pascutti et al. 2017). However, CAD/CAM process is not the only factor that generated superior strength, the material composition and other factors also play important roles. The Vita CAD Temp® consists of acrylate polymer and micro filler reinforced poly-acrylic. Its structure contains a vinyl group with two carbons that difference from other PMMA-CAD/CAM. Surprisingly, strength of acrylate-CAD/CAM was the lowest. This probably related to the structural in-homogeneity as evidence in SEM. The strength behavior of material at lower stress level was precisely described with Weibull analyses, assessing failure probability. The materials with lower σ but high m demonstrated more favorable imparts than a material with high σ but low m, which tended to fail at lower stress levels (Kerby et al. 2013). Resin matrix may degrade due to water absorption from aging, which artificially generated by thermo-cycling from 2,500-10,000 cycles (Kadiyala et al. 2016, Alt et al. 2011). Although, definite protocol for accelerated aging was not established, It was evidenced that thermo-cycling in 5°C and 55°C for 5000 cycles with 30 sec was approximately equal to six months in function of material in the oral cavity (Pascutti et al. 2017, Gülce et al.). Thus the aging process used in this study provides meaningful determination of provisional material for prosthetic dentistry. It was suggested that further study on color stability, marginal and internal accuracy of provisional restorative materials be evaluated upon aging as they are important parameters for validating material in clinical practice.

5. CONCLUSION

Flexural strengths depend upon the difference of provisional restorative materials, which were affected by aging. PMMA-CAD/CAM block possessed highest flexural strength, while Acrylate-CAD/CAM block revealed lowest flexural strength. Bis-acryl resin demonstrated higher flexural strength than Ap-PMMA. The Hp-PMMA provides better strength than Ap-PMMA.

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