

# Effects of surface coating agents on surface microhardness of bis-acryl provisional materials

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**Objective:** To compare surface microhardness of bis-acryl provisional materials after applied various types of surface coating agents using Vickers hardness tester.

**Materials and Methods:** Forty bis-acryl specimens were prepared, polished and randomly divided into 8 groups, with n=5, 1) Protemp<sup>TM</sup>4 without surface coating, 2) Protemp<sup>TM</sup>4 coated with Palaseal<sup>®</sup>, 3) Protemp<sup>TM</sup>4 coated with EQUIA Forte<sup>®</sup> coat, 4) Protemp<sup>TM</sup>4 coated with Optibond<sup>TM</sup> FL, 5) LuxaTemp<sup>®</sup> without surface coating, 6) LuxaTemp<sup>®</sup> coated with Palaseal<sup>®</sup>, 7) LuxaTemp<sup>®</sup> coated with EQUIA Forte<sup>®</sup> coat, 8) LuxaTemp<sup>®</sup> coated with Optibond<sup>TM</sup> FL. The specimens from each group were tested for surface microhardness by Vickers surface microhardness tester. Load used was 50 gf. for 15 seconds. The indentations were evaluated and measured with stereomicroscope at 50x. The measurements were converted into HV (Vickers hardness number) and analyzed with two-way ANOVA.

**Results:** The mean surface microhardness values of LuxaTemp<sup>®</sup> group were statistically higher than Protemp<sup>TM</sup>4 group. The mean surface microhardness values of LuxaTemp<sup>®</sup> group were statistically decreased after application of Palaseal<sup>®</sup> and Optibond<sup>TM</sup> FL. There were no statistically significant difference between the surface microhardness values of LuxaTemp<sup>®</sup> coated with EQUIA Forte<sup>®</sup> coat and LuxaTemp<sup>®</sup> control group. However, it was statistically different from the surface microhardness of the other groups.

**Conclusion:** Three coating agents applied on Protemp<sup>TM</sup>4 have no effects on its surface microhardness. On the other hand, surface microhardness of coated LuxaTemp<sup>®</sup> group showed statistically significant decrease, except for EQUIA Forte<sup>®</sup> coating group which was not statistically significant different from control group. (P<0.05)

**Keywords:** bis-acryl provisional materials, Microhardness, surface coating agent, surface hardness

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## Introduction

Bis-acryl resin composite is the material for temporization that was introduced in 1990s to overcome the negatives of polymethyl methacrylate (PMMA) in terms of mechanical properties, physical properties and application methods[1]. Due to the structure of bis-acryl monomer, it provides crosslinking with another monomer chain that causes high mechanical properties[2,3] compared to the linear structure of polymethyl methacrylate (PMMA)[1,2,4,5]

In the clinical practice, because of the various color shades of bis-acryl resin composite, smoothness and less porous surface compared to methacrylate groups[6], bis-acryl resin composite is often used for esthetic reasons. After polishing, the surface can be coated with surface coating agents to improve its surface qualities by providing better color stability and surface smoothness[7,8] by reducing surface irregularities and fill out their micro-defects and micro-fissures without creating oxygen inhibited layer. Even in areas that are difficult to polish, such as interproximal areas of

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indirect composite restorations and fissures of posterior tooth. There are many studies revealed the advantages of using surface coating agents together with bis-acryl provisional restoration on the esthetics aspect.

However, in the situation of posterior teeth provisional restoration that receive the occlusal loads and exposed to the chemical agents from foods and beverages in the extended period such as in oral rehabilitation case, the mechanical properties of bis-acryl such as flexural strength, surface hardness and wear resistance are needed to be considered. Although several studies compared the mechanical properties of bis-acryl resin composite with other provisional restorative materials, and the results showed that bis-acryl resin composite has higher flexural strength, surface hardness and wear resistance than polymethyl methacrylate (PMMA)[1-3,9]. It is interesting that if the surface coating agents can improve the mechanical properties of bis-acryl material on those properties as well.

Hardness is resistance of material to plastic deformation, this parameter shows the degree of resistance to plastic deformation by an indenter on the material[10]. Surface hardness can be used as an indicator of density, and it can be hypothesized that a denser material would be more resistant to wear and surface deterioration. All provisional materials surface hardness were significantly softened after conditioning in the various concentrations dietary simulating solvents[11] that affect to the occlusal stability and vertical dimension of the provisional restoration because of greater wear. Since there was reported that the coating agents have claimed that it can improve the surface hardness of the PMMA provisional restoration[12]. On the contrary, there was the study showed that surface hardness of composite resins decreased after apply the coating agents[13]. However, the study regarding effects of surface coating agents to surface hardness of bis-acryl resin composite provisional restoration is

still lacking. There are 3 coating agents that used in this study, Palaseal<sup>®</sup>, EQUIA Forte<sup>®</sup> coat and Optibond<sup>™</sup> FL. Palaseal<sup>®</sup> is the coating surface agent that mostly used for PMMA and also bis-acryl while EQUIA Forte<sup>®</sup> coat is the coating for glass ionomer restoration, both of them contain methyl methacrylate as the main component. On the other hand, Optibond<sup>™</sup> FL is the bonding agents for composite filling which contain Bis-GMA as the main component. All coating agents in this study are often and widely used in daily practice. Therefore, the objective of this study was to investigate the difference between surface coating agents on their effects regarding surface hardness of the bis-acryl provisional materials.

## Materials and methods

Provisional material selected were Protemp<sup>™</sup> 4 (3M ESPE, MN, USA.) and LuxaTemp<sup>®</sup> (DMG, Hamburg, Germany.). Surface coating agent selected were Palaseal<sup>®</sup> (Heraeus-Kulzer, Hanau, Germany), Optibond<sup>™</sup> FL (Kerr, CA, USA) and EQUIA Forte<sup>®</sup> coat (GC, IL, USA.).

The split circular shaped stainless steel mold with dimension 10 mm in diameter and 2 mm in height was used for prepare the specimens. [Figure 1] Applying the separating media then the bis-acryl provisional materials were mixed and injected into the mold. The setting times were referred from each manufacturer's instructions. (Protemp<sup>™</sup> 4 for 5 minutes and LuxaTemp<sup>®</sup> for 7 minutes)

All specimens were removed from the mold and immersed in distilled water (37±1 degree Celsius) for 24 hours. Each group of specimens were fixed into PVCs with dental stone type III (5 specimens per PVC).[Figure 2] The fixed specimens were finished and polished to achieve an even plane on every specimens by using a polishing machine (Metaserv 2000, Buehler, USA) and water coolant with the silicon carbide abrasive

paper from 800-grit to 2000-grit (TOA, Samut Prakan, Thailand) respectively. All specimens were rinsed with distilled water, cleaned with ultrasonic for 10 minutes and air dried.

The specimens were randomly divided into 8 groups (n=5), which were 1) Protemp<sup>TM</sup> 4 without surface coating (control group), 2) Protemp<sup>TM</sup> 4 coated with Palaseal<sup>®</sup>, 3) Protemp<sup>TM</sup> 4 coated with EQUIA Forte<sup>®</sup> coat, 4) Protemp<sup>TM</sup> 4 coated with Optibond<sup>TM</sup> FL, 5) LuxaTemp<sup>®</sup> without surface coating (control group), 6) LuxaTemp<sup>®</sup> coated with Palaseal<sup>®</sup>, 7) LuxaTemp<sup>®</sup> coated with EQUIA Forte<sup>®</sup> coat, 8) LuxaTemp<sup>®</sup> coated with Optibond<sup>TM</sup> FL. All coating specimens were applied with a soft brush, in a thin film using only one directional movements on the top surface. Then light cured followed by manufacturer's manual of each surface coating agents (90 seconds for Palaseal<sup>®</sup>, 20 seconds for EQUIA Forte<sup>®</sup> coat, 40 seconds for Optibond<sup>TM</sup> FL). All specimens were immersed in distilled water (37±1 degree Celsius) for 24 hours and gently air dried.

The surface hardness test was conducted with Vickers hardness testing machine (FM-810, FUTURE-TECH, Kanagawa, Japan). [Figure 3] Five indentations, distance 1 mm apart from each indentation, were created on each specimens by a diamond indenter with the load of 50 gf. The load was applied for 15 seconds per interval according to ISO 6507-1[14]. The diagonal ends of the indentation were measured using a stereo-microscope under the magnification of 50x. [Figure 4]



Figure 1 Split circular shaped stainless steel mold.

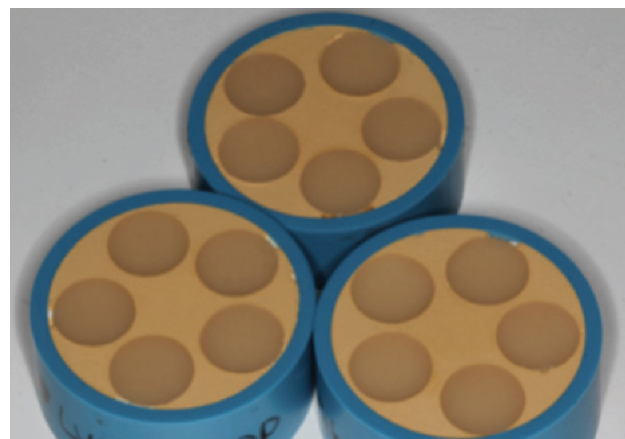


Figure 2 Specimens were mounted into PVC with dental stone type III.

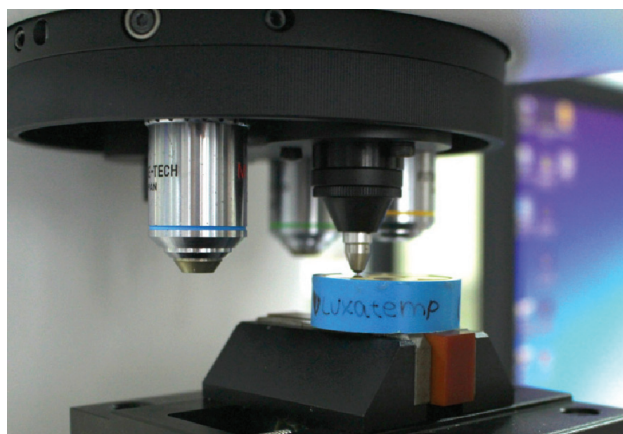
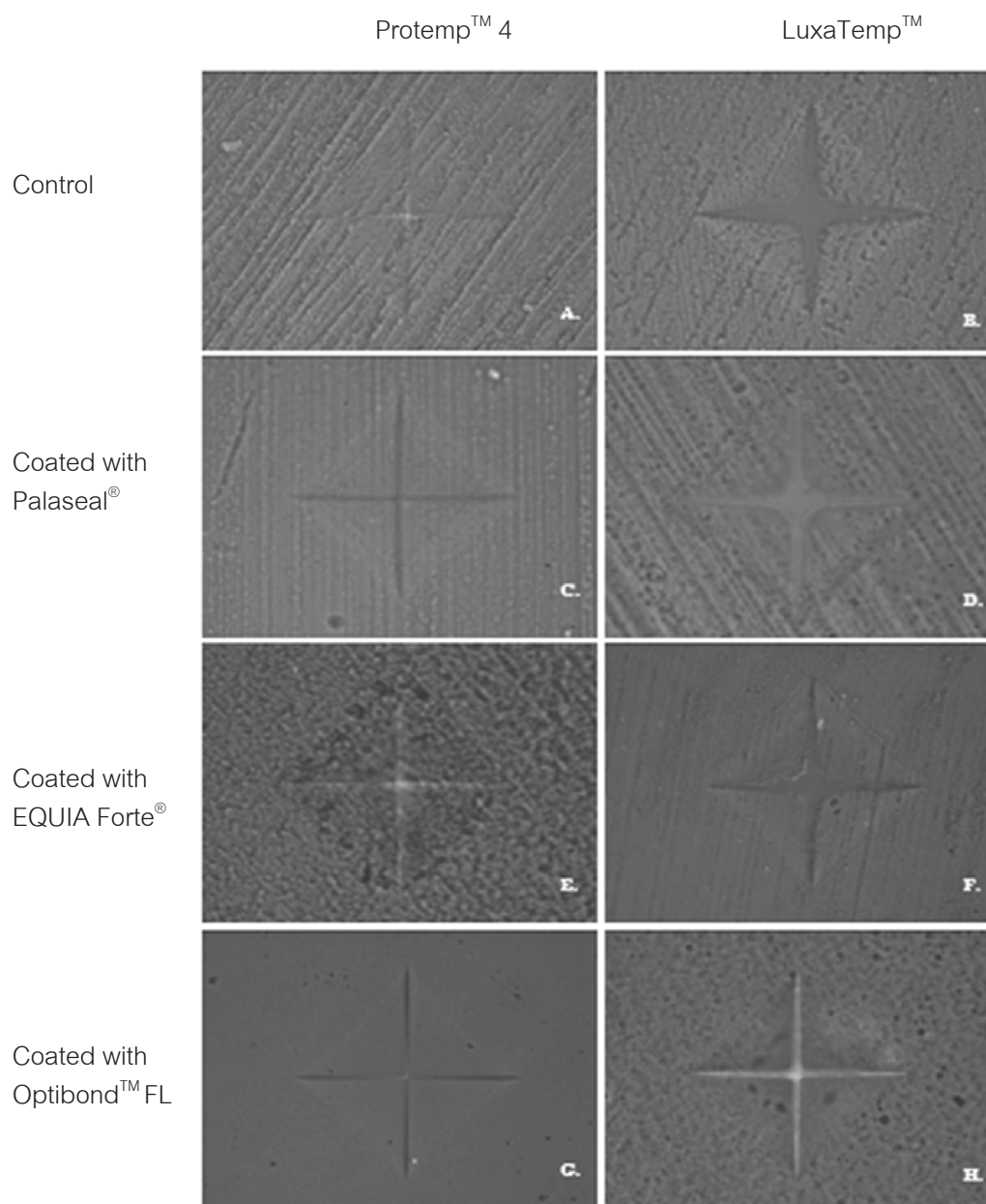


Figure 3 Loading on Vickers hardness tester.



**Figure 4** Stereomicroscope images at 50x magnification of Protemp™ 4 without surface coating (control group)(A), Protemp™ 4 coated with Palaseal® (B), Protemp™ 4 coated with EQUIA Forte® coat (C), Protemp™ 4 coated with Optibond™ FL (D), LuxaTemp® without surface coating (control group)(E), LuxaTemp® coated with Palaseal®(F), LuxaTemp® coated with EQUIA Forte® coat (G), LuxaTemp® coated with Optibond™ FL (H).

## Data analysis

The mean Vickers hardness values were recorded and interpreted using SPSS program version 24.0. The surface hardness value between Protemp™ 4 and LuxaTemp® groups were evaluated with T-test. The surface hardness value between each surface coating agent were

evaluated using one-way ANOVA. The comparisons between both bis-acryl provisional materials with the coating agents were evaluated by two-way ANOVA and tukey HSD post-hoc test.  $P < 0.05$  represents statistically significance. The surface texture after indentation were analyzed by stereo microscope (Olympus stereo Microscopes, ZS61, Nagano, Japan).



## Results

The Vickers surface hardness values of control groups, Protemp<sup>TM</sup>4 group was statistically significant lower from LuxaTemp<sup>®</sup> group. The surface hardness value between surface coating agent groups; Palaseal<sup>®</sup>, EQUIA Forte<sup>®</sup> coat and Optibond<sup>TM</sup> FL. Palaseal<sup>®</sup> were not statistically significant different. After coating with three surface coating materials, surface hardness values of all Protemp<sup>TM</sup>4 groups were slightly decrease but not statistically significant different from control group and the coating groups were not statistically significant different among groups. The surface hardness values of LuxaTemp<sup>®</sup> groups with coating were statistically significantly decrease compared to the control group except the LuxaTemp<sup>®</sup> coated with EQUIA forte<sup>®</sup> group. ( $P < 0.05$ )

## Discussion

Surface hardness is a good indicator of resistance to wear and surface deterioration. Surface hardness test can be carried out by Vickers hardness tester and Knoop hardness tester. The Vickers hardness tester can be used on every types of materials[12]. Vickers hardness tester is also more useful than Knoop hardness in this study because square shaped indenter of Vickers hardness tester is more structure conservation than Knoop hardness tester[2].

In this study, comparison between control specimens of each bis-acryl provisional material were made. LuxaTemp<sup>®</sup> (mean Vickers hardness value  $23.50 \pm 0.71$ ) showed superior surface hardness compared to Protemp<sup>TM</sup>4 (mean Vickers hardness value  $18.75 \pm 0.88$ ). [Table 2.] The manufacturer data sheets report that the matrix of LuxaTemp<sup>®</sup> has 44% by weight glass filler particles, while Protemp<sup>TM</sup>4 has only 20-30%. The filler

contents may contributed to the statistically significant higher value in surface hardness in the LuxaTemp<sup>®</sup> than Protemp<sup>TM</sup>4 groups. Size of filler particles is another factor that may effects surface hardness difference between Protemp<sup>TM</sup>4 and LuxaTemp<sup>®</sup>. Protemp<sup>TM</sup>4 filler particles resemble that of nanofilled composite with sizes between 20-30 nm and dispersed spherical particles in the range of 40–50 nm while LuxaTemp<sup>®</sup> filler particles size are up to 3  $\mu\text{m}$ . Microhybrid nature of LuxaTemp<sup>®</sup> makes surface hardness of LuxaTemp<sup>®</sup> higher than Protemp<sup>TM</sup>4.

In the comparison of the specimens after application of surface coating agents. One-way ANOVA analysis showed the Vickers hardness values after application of surface coating agents were decrease compared to the control group but not statistically significant different among groups. ( $P < 0.05$ ) (mean Vickers hardness value  $21.12 \pm 2.61$ ,  $18.13 \pm 1.46$ ,  $20.17 \pm 2.16$ , and  $17.84 \pm 1.28$  for control group, Palaseal<sup>®</sup>, EQUIA Forte<sup>®</sup> coat, and Optibond<sup>TM</sup> FL groups respectively) [Table 3.] These results might be explained by the effects of oxygen inhibited layer on surface of bis-acryl resin material in the same way as applying the surface coating agents on resin composite filling materials[13] because of the similar component of bis-acryl material and resin composite filling material. Although in our study, formation of the inhibited layer was prevented by light-curing under a glass slide, but still, the results showed the decreasing of surface hardness. Palaseal<sup>®</sup> is the coating surface agent that mostly used for PMMA and also bis-acryl while EQUIA Forte<sup>®</sup> coat is the coating for glass ionomer restoration, both of them contain methyl methacrylate as the main component. On the other hand, Optibond<sup>TM</sup> FL is the bonding agents for composite filling which contain Bis-GMA as the main component. The results showed that either the coating agents that contain methyl methacrylate or Bis-GMA decreased surface hardness of bis-acryl materials. The Optibond<sup>TM</sup> FL group showed

the lowest surface hardness. This could be described from the Table 1. that there is the ethanol in Optibond™ FL which cause the soften surface hardness of bis-acryl restoration material

as reported in previous study[11]. This also confirm the effect of the ethanol on softening the surface of bis-acryl material.

**Table 1** The composition of materials used in this study.

Material	Type	Composition	Lot number	Manufacturer
Protemp™4	autopolymerized	2,2'-[(1-methylethylidene)bis(4,1-phenyleneoxy)] bis-, diacetate, benzyl-phenyl-barbituric acid, silane treated silica, tert-butyl peroxy-3,5,5-trimethylhexanoate, silane treated silica, dimethacrylate	322956	3M ESPE, MN, USA
LuxaTemp®	autopolymerized	Ethoxylated bisphenol-A dimethacrylate, polyester, UDMA, TEGDMA, barium glass, pyrogenic silica	780514	DMG, Hamburg, Germany
Palaseal®	Light-cured 20 seconds	Methyl methacrylate, 2-hydroxyethyl isocyanuratetriacrylate, oligotriacrylat and diphenyl(2,4,6-trimethylbenzoyl)phosphinoxid	10132	Heraeus-Kulzer, Hanau, Germany
EQUIA Forte® coat	Light-cured 20 sec-onds	Methyl methacrylate, photoinitiator, synergist, phosphoric acid ester monomer, butylated hydroxytoluene	1612061	GC, IL, USA.
Optibond™ FL	Light-cured 40 sec-onds	Bis-GMA, HEMA, GPDM, ethanol, barium-aluminum borosilicate glass, disodium hexafluorosilicate, fumed silica (24% filler)	5344022	Kerr, CA, USA.

**Table 2** Mean and standard deviation Vickers hardness values of all groups. (n=5) Superscript letters A and B indicate statistical significance in VHN among groups. (P<0.05)

	Control	Palaseal®	EQUIA Forte® coat	Optibond™ FL
Protemp™ 4	18.75±0.88 <sup>A</sup>	18.34±2.07 <sup>A</sup>	18.45±1.17 <sup>A</sup>	17.01±0.89 <sup>A</sup>
LuxaTemp®	23.50±0.71 <sup>B</sup>	17.92±0.53 <sup>A</sup>	21.9±1.31 <sup>B</sup>	18.66±1.10 <sup>A</sup>

**Table 3** Mean and standard deviation Vickers hardness values of groups divided by type of surface coating agents. (n=10) Superscript letters a and b indicate statistical significance in VHN among groups. (P<0.05)

	Mean±SD
Control	21.12±2.61 <sup>a</sup>
Palaseal®	18.13±1.46 <sup>a</sup>
EQUIA Forte® coat	20.17±2.16 <sup>a</sup>
Optibond™ FL	17.84±1.28 <sup>a</sup>

However, in LuxaTemp<sup>®</sup> groups, the Vickers hardness after application of Palaseal<sup>®</sup> and Optibond<sup>™</sup> FL were statistically significant decreased compared to the control group. (mean Vickers hardness value  $17.92 \pm 0.53$ , and  $18.66 \pm 1.10$  for Palaseal<sup>®</sup> and Optibond<sup>™</sup> FL respectively). While after application of EQUIA Forte<sup>®</sup> coat, the surface hardness was not statistically significant different from the control group. (mean Vickers hardness value  $21.90 \pm 1.31$ ) EQUIA Forte<sup>®</sup> coat surface coating agent is a part of EQUIA Forte<sup>®</sup> system, its original purpose was to be a surface coating for glass ionomer cement restorative material. EQUIA Forte<sup>®</sup> coat was evaluated and compared with other surface coating agents by its manufacture on glass ionomer cement restorative material and showed lower depth of wear and higher hardness number compared to other surface coating agents[15]. However, there is no study that evaluate surface hardness of EQUIA Forte<sup>®</sup> coat on other materials to correlate this study with. After Vickers hardness testing, none of the specimens in bis-acryl provisional materials showed increased surface hardness value. However, EQUIA Forte<sup>®</sup> coated on LuxaTemp<sup>®</sup> specimens also showed the decreasing of surface hardness value compared to control group but not statistically significant. ( $p < 0.05$ ) During experiment, it shows that light-cured surface coating agent revealed oxygen-inhibiting layer, which was observed as shiny surface that showed scratch lines after scratching. Oxygen-inhibiting layer was formed during the polymerization of the resins, diffusion of oxygen into the resin inhibits the polymerization reaction by forming peroxide radicals. An unreacted double bond or a free monomer layer in the surface will remain on the surface as oxygen-inhibiting layer. It has several effects on prognosis of the restorations; reducing hardness, wear resistance and marginal adaptation[10,15].

Among all the coated specimens, Luxatemp<sup>™</sup> coated specimens showed visible oxygen-inhibiting layer except specimens coated with EQUIA Forte<sup>®</sup> coat, which the least oxygen-inhibiting layer was observed. This could be the reason that Vickers hardness number of Luxatemp<sup>™</sup> after EQUIA Forte<sup>®</sup> coat application was not statistically significant decreased. Oxygen-inhibiting layer can be eliminated by alcohol application after light curing had been done. However, due to the manual, none of surface coating agents used in this study recommended the elimination oxygen-inhibiting layer by alcohol application.

After application of surface coating agents on Protemp<sup>™</sup>4 specimens, the Vickers hardness number of all the coated specimens were not statistically significant different. It could be described that according to the manufacturer's manual, application of surface coating agents should be applied as a thin film covering its surfaces. Possibly, the indenter could pass through the thin film layer and measured the underlying Protemp<sup>™</sup>4 specimens directly. As a result, the Vickers hardness number of the coated specimens in Protemp<sup>™</sup>4 group was not significantly different compared to the control group.

After the surface hardness evaluations were made, the study concluded that application of three surface coating agents (Palaseal<sup>®</sup>, EQUIA Forte<sup>®</sup> coat and Optibond<sup>™</sup> FL) the surface hardness of Protemp<sup>™</sup>4 are not statistically significant different. On the other hand, the surface hardness of Luxatemp<sup>™</sup> were statistically significant decreased by applying with Palaseal<sup>®</sup> and Optibond<sup>™</sup> FL. ( $P < 0.05$ ) The data reported provide guidance for clinicians to consider the advantages of applying surface coating agent on bis-acryl provisional resin materials in esthetics and function benefits.

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