The Effect of Addition Nanoparticles Kraft Lignin to the Acrylic-based Provisional Restorations Crown and Bridge

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ABSTRACT

Aim and objective: To evaluate the effect of adding nanoform of biopolymer kraft lignin (KL) as reinforcement material as it had many functional groups to the acrylic-based provisional restorations crown and bridge for improving their properties.

Materials and methods: The specimens were grouped according to the powder of nano KL which was added to the acrylic resin after sonication with the methyl methacrylate monomer in percentages of 0.5, 0.75, and 1.0% to be in four groups with the control without addition. In total, 112 specimens (28 per each test) were cured and fabricated to evaluate some mechanical properties, for transverse strength test, hardness test, surface roughness, and specimen for impact strength test, results were analyzed using Statistical Package for the Social Sciences.

Results: Highly significant increase was obtained (p < 0.05) among the groups, in transverse strength (87.3243 N/mm²); impact strength (9.1071 kJ/m²) at 1.0 wt% group, which is the highest value; and hardness number concentration of KL 1.0% which had the highest mean value (84.0943); while the result indicates an increase in the surface roughness values than the control.

Conclusion: Using such material KL in nanoform has a positive effect on the mechanical properties which are tested in acrylic used for crown and bridge, especially in the (1.0%) addition KL further study could be done for higher percentage.

Clinical significance: According to this novel addition which indicated that lignin as a nanoparticle may have had interaction among the polymer matrix. So, it may anticipate in improving the mechanical properties by increasing the strength of the cold-cured acrylic resin.

Keywords: Kraft lignin, Nanoparticles, Polymethyl methacrylate.

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INTRODUCTION

Provisional crown and bridge replacements are commonly used for days to weeks, necessitating that they be well made, stable, and serve distinct functions and purposes. They serve as an important diagnostic and assessment tool for effective function, color, form, contour, occlusion, periodontal response, implant healing, and most importantly, esthetics and good protection for teeth.^{1–3}

Polymethyl methacrylate (PMMA) has been commonly used in the construction of provisional fixed partial dentures in use. Recent years have seen the introduction of poly(ethyl methacrylate), polyvinyl(ethyl methacrylate), bis-acrylic composite resin, visible light-cured urethane dimethacrylate, and microfilled.^{4–6}

Provisional restoration discoloration can be an esthetic issue, particularly if the treatment plan is needed for long-time use.⁷ Acrylic resins are quickly worn off which was improved in part by the use of filling materials.⁸

Polymethyl methacrylate has several flaws that should be improved, such as the mechanical properties of these polymers. In the first three years of use, more than 60% of dental prostheses were broken.⁹

Copolymerization and creation of blends or composites based on polyacrylates may enhance their properties (MMA). Addition of different organic or inorganic compounds during the polymerization process had an effect on the properties of the acrylic resins.¹⁰

It was proved that the addition to the resin based on bisphenol A-glycidyl methacrylate inorganic fillers of micrometric size causes a significant improvement in the strength properties. The same improvement by introducing inorganic fillers having a nanosize to the matrix based on poly(methyl methacrylate).^{11,12}

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The filler organic, inorganic, and metallic particulate materials in both micro and nanosizes which reinforce polymers and polymer matrix composites have a wide range of uses.^{13,14}

The complexity structure of the lignin makes it hard to isolate a selected product and also the effects of pretreatments thereon, therefore to know lignin polymeric properties and linkages it needs vast characterization to identify and extract from it, as well as the properties of the functional groups connected to the aromatic ring.¹⁵ Owing to the inclusion of phenolic and aliphatic hydroxyl groups in its structure, lignin is an excellent candidate for the production of new materials. These give the macromolecule a lot of chemical modification potential.¹⁶

Alkali lignin is the most product lignin class. It is a by-product of the biofuel and paper industries that is isolated from fibers by chemical pulping, but has a lower value than the primary products.¹⁷ It has many functional groups which may interact with matrix making composite or copolymer and in nanoform which

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has good surface energy that may result in good properties, the chemical structure of KL can be seen in Figure 1. Ismail and Ismail et al. studied the effect of lignin when added to the transverse and impact strength of the autopolymerizing acrylic resin, during this study improvement of these properties has been found.^{18,19} So according to the results of these studies the decision was made to use it, as it has many functional groups which may interact with the matrix of crown and bridge restorative material and make changes in the mechanical properties for the best results.

MATERIALS AND METHODS

The materials used in this study: cold-curing PMMA especially for crown and bridge (powder and liquid, Major Italy), alkali KL (powder, Aldrich company) used as reinforced material.

Nanolignin Preparation

The KL was crushed from the micro powder received from the company to nanosize particles 30–40 nm in the laboratories of Ministry of Science and Technology using high energy ball milling type (QM-IP04) for 20 hours. Figure 2 shows that the lignin has dense aggregates with sizes higher than 0.51 µm; on the contrary, the lignin showed a particle crystalline-like structure.

Method of Adding Lignin

The nanofiller lignin was in the monomer dispersed using ultrasonic running at 120 W 60 kHz with a period of 180 seconds to prevent aggregation and then nanolignin was mixed with PMMA polymer, and proceeded in normal flasking method to get the suitable test samples. The nanoparticle lignin was added to the acrylic resin of 0.5, 0.75, and 1.0%. A total of 112 specimens (28 for each test in all percentages) were used, as seven specimens for each of the selected group, as a result having four groups; one control without addition with other three ones.

Mold Preparation

Plastic pattern blocks were used to prepare molds with specific measurement sizes assigned by computer. Plastic blocks of dimensions $65 \times 10 \times 2.5$ mm were prepared for transverse strength, hardness, and surface roughness test while specimens of $80 \times 10 \times 4$ mm (ISO 179,

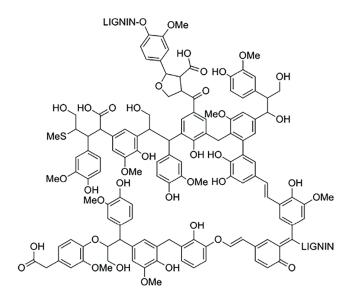


Fig. 1: Structure of alkali lignin

2000) for unnotched specimens were prepared for impact strength test. The plastic specimens were immersed in the stone slurry. Then continued as conventional flasking method. Powder\liquid ratio of acrylic was used according to the manufacturer's instruction. The flasks were clamped and then put in curing path for 30 minutes at 40°C according to a study.¹⁸ After finishing, the acrylic specimens were carefully removed. Excess materials were removed and the specimens got polished and finished except for the surface roughness which was left without polish. This is for the control group while for the other groups the percentage weight of nano KL of 0.5, 0.75, and 1.0% each percent subscribed from the powder weight then dispersed in dispersing apparatus for 3 minutes in the monomer and immediately then mixed with the powder of polymethyl methacrylate, wait for dough stage then cured as the same manner for all specimens. All specimens were conditioned for 48 hours in filtered water at 37°C before being tested in accordance with American Dental Association standard number 12.20

Transverse Strength Test

The transverse strength test was achieved using universal testing equipment by Instron (WDW-200E), each specimen was put on a bending fixture made of two straight supports with 50 mm between them, the scale was 50 kg and the load was given with head speed of 2 mm/min by a bar placed in the center between the supports, causing deflection until fracture happened.

$$S = \frac{3FI}{2bd^2}$$

where, S: transverse strength F: maximum load in Newton I: supporting width in mm d: height of the specimen in mm b: width of the specimen in mm.

Impact Strength Test

The specimens were supported vertically by Izod impact test and struck by free-swinging pendulum of 5.5 J. The scale readings give the impact energy absorbed in Joule during breaking. The Izod impact strength of unnotched specimens was calculated in kiloJoule per square meter by the following equation:

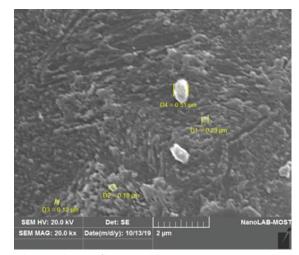


Fig. 2: The particle size of lignin and their aggregation were determined using scanning electron microscopy

The strength of impact = (ISO 179, 2000) in kJ/m^2

$$e = \frac{e}{db} 10^3$$

where, *e*: energy, *d*: depth, *b*: width.

Surface Hardness Test

With dimensions of $65 \times 10 \times 2.5 \pm 0.1$ mm, the acrylic resin specimens were prepared, Shore D durometer hardness tester was used to determine the hardness of the acrylic resin substance. The device consists of an indenter with a diameter of 0.8 mm and a digital scale with a range of 0–100 units. The procedure entails pressing down firmly and quickly on the indenter and taking a reading. Three readings were taken on each specimen (one in the middle and one at each end), and the mean of these readings was calculated.

Surface Roughness Test

Specimens with $65 \times 10 \times 2.5 \pm 0.1$ mm in dimensions were manufactured for this test. The profilometer was used to evaluate the effect of nanoparticles reinforcement on the test surface's microgeometry. This tool comes with a diamond-tipped sharp stylus surface analyzer that can trace the profile of surface abnormalities by recording all of the peaks and recesses that define the surface's scale. In its stable stage, the acrylic was placed and the region of the test was chosen as it separated into four pieces, after which the analyzer was then moved along the tested area, and the mean of four records was calculated.

Statistical Analysis

Statistical Package for the Social Sciences was used for statistical analyses which includes descriptive and statistical tables. Analysis of variance (ANOVA) was done for the results to check the significant difference among the groups. The least significant difference (LSD) test was used to determine the statistical significance of the mean difference between control and each group.

Results

Transverse Strength Test

The mean value of transverse strength had shown that 1.0% was the highest value within the tested groups of transverse strength test as shown in Table 1 and Figure 3.

The differences between the control and other groups with multiple comparisons LSD test were analyzed and it showed a highly significant difference revealed between control and all other groups as shown in Table 2.

Impact Strength Test

The results were revealed (9.1071 kJ/m²) at 1.0 wt% group which was the highest value as shown in Table 3 and Figure 4, ANOVA test between groups showed a highly significant difference.

The higher impact strength was at 0.75 and 1.0 wt% groups than control. The differences between control and all other groups with multiple comparisons LSD test were analyzed and it showed no significant difference in all groups except 1.0 wt% which showed a highly significant difference as shown in Table 4.

Indentation Hardness Test

Measurement of the hardness initially gave some indication of the wear resistance. The Shore "D" hardness number was directly related to the indentation hardness of the tested material. The results which are shown in Table 5, indicated that indentation resistance increased with the group of KL 1.0 wt% which had the highest mean value (84.0943) while the lowest for KL modified PMMA 0.50 wt% value (81.9957) followed by 0.75 wt% which had (83.2857) indentation hardness number as shown in Figure 5. Analysis of variance test revealed that there were highly significant differences in hardness between groups.

The differences between each group and the other one with multiple comparisons LSD test were analyzed and it showed

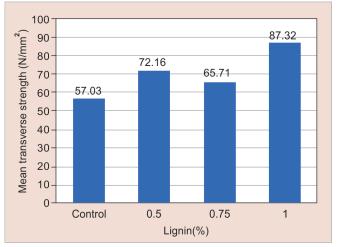


Fig. 3: Histogram of transverse strength mean values of groups

Groups	Ν	Mean	Standard deviation	Standard error	Minimum	Maximum
Control PMMA	7	57.0300	2.77935	1.05050	53.63	61.24
0.5% lignin	7	72.1586	6.69956	2.53219	61.20	78.33
0.75% lignin	7	65.7143	3.50020	1.32295	58.80	69.60
1.0% lignin	7	87.3243	4.55260	1.72072	81.41	93.62
ANOVA test for						
transverse streng	th	Sum of squares	df	Mean square	F	Sig.
Between groups		3430.970	3	1143.657	53.450	.000

 Table 1: Descriptive statistics of transverse strength (N/mm²) and ANOVA test



The Effect of Addition	Nanoparticles	Kraft Lignin to	D PMMA

Table 2: Multiple compression LSD test for transverse strength (N/mm ²) results

(I) Groups	(J) Lignin	Mean difference (I–J)	Standard error	Sig.
Control	0.5%	-15.12857*	2.47251	0.000
	0.75%	-8.68429*	2.47251	0.002
	1.0%	-30.29429*	2.47251	0.000
0.5%	Control	15.12857 [*]	2.47251	0.000
	0.75%	6.44429 [*]	2.47251	0.015
	1.0%	-15.16571*	2.47251	0.000
0.75%	Control	8.68429 [*]	2.47251	0.002
	0.5%	-6.44429 [*]	2.47251	0.015
	1.0%	-21.61000*	2.47251	0.000
1.0%	Control	30.29429 [*]	2.47251	0.000
	0.5%	15.16571 [*]	2.47251	0.000
	0.75%	21.61000*	2.47251	0.000

*p = <0.05

Table 3: Descriptive statistics of impact strength (kJ/m²) and ANOVA test

Groups	Ν	Mean	Standard deviation	Standard error	Minimum	Maximum
Control PMMA	7	6.7143	0.56695	0.21429	6.25	7.50
0.5% lignin	7	6.8929	0.62678	0.23690	6.25	7.50
0.75% lignin	7	7.0714	0.55367	0.20927	6.25	7.50
1.0% lignin	7	9.1071	0.60994	0.23053	8.75	10.00
ANOVA test for impact	strength					
(kJ/m ²)	_	Sum of squares	df	Mean square	F	Sig.
Between groups		26.188	3	8.729	25.068	.000

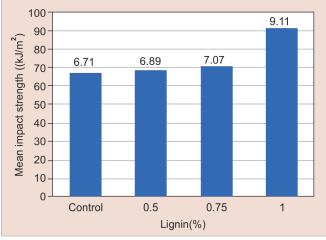
Table 4: Multiple comparisons LSD test for impact strength (kJ/m²)

(I) Groups	(J) Lignin	Mean difference (I–J)	Standard error	Sig.
Control	0.5%	-0.17857	0.31542	0.577
	0.75%	-0.35714	0.31542	0.269
	1.0%	-2.39286*	0.31542	0.000
0.5%	Control	0.17857	0.31542	0.577
	0.75%	-0.17857	0.31542	0.577
	1.0%	-2.21429 [*]	0.31542	0.000
0.75%	Control	0.35714	0.31542	0.269
	0.5%	0.17857	0.31542	0.577
	1.0%	-2.03571*	0.31542	0.000
1.0%	Control	2.39286*	0.31542	0.000
	0.5%	2.21429 [*]	0.31542	0.000
	0.75%	2.03571*	0.31542	0.000

**p* = <0.05

Table 5: Descriptive statistics of hardness (No.) and ANOVA test

Groups	Ν	Mean	Standard deviation	Standard error	Minimum	Maximum
Control PMMA	7	81.8914	0.38925	0.14712	81.33	82.57
0.5% lignin	7	81.9957	0.67508	0.25516	81.30	82.67
0.75% lignin	7	83.2857	0.35636	0.13469	82.67	83.67
1.0% lignin	7	84.0943	0.24946	0.09429	83.67	84.33
ANOVA test hardness (No.))	Sum of squares	df	Mean square	F	Sig.
Between groups		23.676	3	7.892	39.636	.000



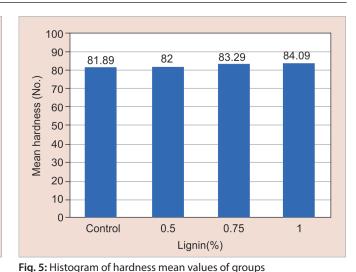


Fig. 4: Histogram of impact strength mean values of groups



(I) Lignin	(J)	Mean difference (I–J)	Standard error	Sig.
Control	0.5%	-0.10429	0.23852	0.666
	0.75%	-1.39429*	0.23852	0.000
	1.0%	-2.20286*	0.23852	0.000
0.5%	Control	0.10429	0.23852	0.666
	0.75%	-1.29000*	0.23852	0.000
	1.0%	-2.09857 [*]	0.23852	0.000
0.75%	Control	1.39429*	0.23852	0.000
	0.5%	1.29000*	0.23852	0.000
	1.0%	-0.80857*	0.23852	0.002
1.0%	Control	2.20286*	0.23852	0.000
	0.5%	2.09857*	0.23852	0.000
	0.75%	0.80857*	0.23852	0.002

*p = <0.05

Groups	Ν	Mean	Standard deviation	Standard error	Minimum	Maximum
Control	7	2.0096	0.27153	0.10263	1.67	2.32
0.5%	7	3.9587	0.13346	0.05044	3.84	4.21
0.75%	7	3.1541	0.37469	0.14162	2.83	3.95
1.0%	7	3.8693	0.40267	0.15219	3.34	4.33
ANOVA test surfa roughness	се	Sum of squares	df	Mean square	F	Sig.
Between groups	;	17.035	3	5.678	57.639	0.000

highly significant differences revealed in all groups except 0.5 wt% (sig. 0.666) as shown in Table 6.

DISCUSSION

Nanoparticles from lignin have been used as nontoxic agent,²¹ which is one of the most present biopolymers in nature, biocompatible and has good stability in contrast to many other nanoparticles used.²² Trials for new applications of lignin need additional use to them.²³ Lignin has both thermoplastic and thermosetting properties due to its amorphous chemical structure.²⁴ The new development toward its potential use in polymer materials, making micro and nanostructured lignin-based materials are then addressed.²⁵

This study has been done for evaluating the effect of biopolymer lignin as nano KL to the self-curing acrylic resin of

Surface Roughness Test

Resin specimens exhibited surface roughness (Ra = 2.0096 μ m) compared to modified acrylic resin (Ra = 3.8693 μ m) in 1.0 wt% specimens as shown in Table 7 and Figure 6. Analysis of variance test revealed highly significant differences between groups.

The differences between each group and the other with multiple comparisons LSD test were analyzed and it showed a highly significant difference revealed in groups 0.5, 0.75, and 1.0 wt% as shown in Table 8.



provisional restorations crown and bridge material. However, no other study evaluates this material in such dental usage.

Natural polymers contain more functional groups and links lead to choose them. The interaction of reinforcing and plasticization in the action of KL addition is complex.

The size of the lignin molecules and the cross-linked density of the matrix polymer determine the level of development. Mechanical properties were determined to have only a little positive or negative effects as a result of the application. To certain lignin load an improvement to mechanical property was obtained.²⁵

The aromatic structure of lignin provides a rigid component to polymeric systems forming high Tg materials, and its phenolic as well as aliphatic hydroxyl functionalities could be used as chemical handles in many applications.²⁶

According to the results of this study the mechanical properties which include transverse strength, impact strength, surface hardness, and surface roughness have been studied after addition of different percentages of nano KL (0.5, 0.75, and 1.0%) depending on the results of other studies using micro KL^{18,19} which they found best results in 0.5–1.0%, in this study using nanoparticles which has the characteristics of lignin have variant functional groups, for instance hydroxyls, carbonyls, and methoxyls which are active sites for additional chemical variation of lignin. Lignin is a crosslinked polymer which provides a great potential to be employed in the

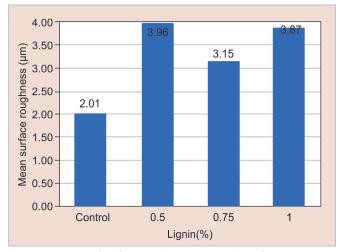


Fig. 6: Histogram of surface roughness mean values of groups

Table 8:	Multiple	comparison	s LSD for	surface rou	ghness (µm	1)

synthesis of materials that are recognized for their interesting applicability in biomedicine.²⁷

The size of the filler particles, as well as their shape and distribution in the polymer matrix and the strong adhesion at the interface, had a more focused effect on the mechanical properties of particulate-filled polymer composites.²⁸ The size of particles needs to be very fine for good processing.²⁹ The effect of nanoparticles in case of low percentages, has good dispersion as well as has a very small size which gives good results.³⁰

In this study, the transverse strength had increased gradually as the amount of nanoparticles KL added to the professional crown and bridge acrylic increased as shown in Table 1 and Figure 3, the maximum increase and highly significant was found in 1.0%, only in 0.75 there was a decrease than the 0.5% which may be related to not good dispersion of the material in the resin matrix and to the high molecular weight of lignin, this acts as not a good compatibilizer, results to decrease the adhesion between PMMA matrix and lignin, and stresses concentration in some areas of nanoparticles and maybe because of the presence of crack propagations in the specimens which is agreed with other.³¹ While this increase is related to good adhesion between the particles and the chemical composition of the resin matrix with very good dispersion of this material and active groups contain which make good results. The transverse strength of the denture base materials can be an important indicator of their performance.³² This study was designed to improve the mechanical properties of PMMA by incorporation of different percentages of nanoparticles of KL. These results agreed with previous studies which tested flexural strength after addition of different nanoparticles SiO₂ and ZrO₂-PMMA composites using such techniques.^{33,34}

Impact strength test is considered to test the resistance to fracture of material to the sudden application of a load.^{17,35} However, depending on the loading configuration (Izod or Charpy), the specimen's dimensions, and the presence of notches and their geometry, this test can result in different values of impact strength. The impact strength increased with increasing nano KL content up to 1.0 wt% (9.1071 kJ/m²), as shown in Table 3. This inclusion of nano KL resulted in a plasticization effect; plasticization is known to have a beneficial effect on the fracture properties of polymer. It is common practice to add plasticizers to these polymers to improve their sharp fracture properties. This is in agreement with other studies on micro particles of lignin.²⁶

(I) Lignin	(J)	Mean difference (I–J)	Standard error	Sig.
Control	0.5%	-1.94914*	0.16777	0.000
	0.75%	-1.14457*	0.16777	0.000
	1.0%	-1.85971*	0.16777	0.000
0.5%	Control	1.94914*	0.16777	0.000
	0.75%	0.80457*	0.16777	0.000
	1.0%	0.08943	0.16777	0.599
0.75%	Control	1.14457*	0.16777	0.000
	0.5%	-0.80457*	0.16777	0.000
	1.0%	-0.71514*	0.16777	0.000
1.0%	Control	1.85971*	0.16777	0.000
	0.5%	-0.08943	0.16777	0.599
	0.75%	0.71514*	0.16777	0.000

*p = <0.05

The highly significant increased value in impact strength may be related to the effect of interaction between nanoparticles KL and PMMA polymer depending on the size of the specific surface area of the filler, smaller particles have larger surface area; leading to increase in strength. This is agreed with Tanasă et al.³⁶ Also, this increase in value may be due to the interfacial shear strength between matrix and nanofiller which is high due to the formation of supramolecular bonding or cross-links which shields the nanofillers which in turn inhibits cracks propagation. This is also suggested by Sun et al.³⁷ The addition of nanofiller may form efficient network (three-dimensional network) of PMMA and lignin nanoparticles especially because lignin has many functional groups. Polymethyl methacrylate chain totally transferred into 3D network-like chains at 1% of nanofiller thus leads to a decrease in the segmental motion and an increase in the impact strength.

It has been stated that surface hardness of composite resins is influenced by both organic matrix and fillers. With regard to the organic matrix, hardness depends on the density and structure of the polymer formed and degree of conversion after polymerization.³⁸ It was found in this study that hardness value showed a highly significant increase in the studied groups compared with control group as shown in Table 5. The increased hardness of the nanocomposite at 1 wt% may be attributed to the accumulation of the KL nanoparticles into the acrylic matrix especially on the surface. Similar findings were reported by Al-Hiloh and Ismail who add ZrO₂ and Ahmed and Ismail who added SiO₂.^{33,34} The presence of improvement in this property may be due to lack of clustering of the particles within the resin, which can have a weakening effect on the resin.³⁹ which reported increase in hardness number by adding TiO₂, ZrO₂ inorganic particles. Also, these results agreed with the study of Ismail who used the same material but micro added to autopolymerizing resin with modified curing environments and found that hardness number (84.5250) found in 1.0 wt% curing at 40°C.¹⁸

As for surface roughness, comparisons of Ra values with other studies cannot be done because of differences in the experimental techniques, procedure used for polishing as well as measuring the surface roughness, and differences in the type of materials used. It appears from the literature that the roughness of dental acrylic resins is mainly affected by material inherent features and polishing procedure, they found that Ra of autopolymerizing resin is 0.36 µm when using pumice slurry and 0.10 µm when using universal polishing paste. The surface roughness is significantly reduced by polishing procedures, so in this study we do not polish the samples as this may lead to increase in the surface roughness. The increase in the roughness of the resin may be attributed to the stress at the filler-matrix interface. As a consequence, the filler particles located at the surface of the material would be deboned and the grooves created would promote the increase in the roughness, as observed in this study, the materials evaluated in this study may be considered with high roughness according to when the materials' roughness ranged from 0.7 to 3.4 µm, this is because of no usage of any polishing agent which makes it more smooth.40

CONCLUSION

With the limitation of this study, using such material KL in nanoform has shown a positive effect on the mechanical properties which are tested in acrylic used for crown and bridge especially in the (1.0 %) which enhances the clinical use by increasing the strength

of this material, additionally, further study could be done for a higher percentage.

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