



Surface Roughness, Hardness and FTIR of the Modified Soft Liner with Different Metal Oxide Nanoparticles

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Article information

Received: 20 April, 2020

Accepted: 20 June 2021

Available online: 10 September, 2022

Keywords:

Metal oxide nanoparticles.

Soft liner.

Shore A.

Roughness.

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Abstract

Aims: To study the effect of metal oxide nanoparticles (MgO, ZrO₂, ZnO) on acrylic based soft liner surface properties and FTIR. **Materials and Methods:** Acrylic-based soft-liner specimens were prepared by adding three different concentrations (0.5, 1, and 2) %wt of metal oxide nanoparticles (MgO, ZrO₂, ZnO). A disk-shaped specimens with 30mm diameter and 3mm thickness were prepared for shore (A) hardness test to evaluate the surface hardness of modified soft liner. The surface roughness of soft-liner samples (10X10X2mm) was tested by a profilometer. FTIR analysis was conducted to evaluate chemical reaction that may occur between acrylic-based soft liner and nanoparticles. **Results:** there were no chemical reaction carried out between soft liner and metal oxide nanoparticles at different concentration- surface hardness (shore A) was increased as nanoparticles concentration increased in modified resin. Nanoparticles with 1 and 2% concentration incorporated in soft liner had lower surface roughness value. **Conclusions:** Metal oxide nanoparticles have improved the surface texture of acrylic-based soft liner, while the hardness of modified soft liner was increased with nanoparticles concentration increases.

الخلاصة

الأهداف: تهدف الدراسة الى دراسة تأثير الجسيمات النانوية لأكاسيد معادن (ZnO، ZrO₂، MgO) على خصائص سطح بطانة الطقم المرنة ذات الاساس الاكريليك. **المواد وطرائق العمل:** تم إعداد عينات بطانة المرنة الأكريليك بإضافة جسيمات نانوية لأكاسيد المعادن (ZnO، ZrO₂، MgO) وبثلاثة تراكيز مختلفة (0.5، 1، 2) % من الوزن). تم تحضير النموذج على شكل قرص وبقطر 30 ملم وبسمك 3 ملم لاختبار صلابة الاسطح (shore) لتقييم صلابة سطح بطانة الطقم المرنة المعدلة. فحصت خشونة سطح عينات بطانة الطقم المرنة بقياسات (10 X 10 X 2 ملم) باستخدام مقياس تباين الأسطح. وقد أجري تحليل FTIR لتقييم التفاعل الكيميائي الذي قد يحدث بين بطانة الطقم المرنة الاكريلية والجسيمات النانوية. **النتائج:** لا يوجد تفاعل كيميائي بين بطانة الطقم المرنة وجسيمات نانوية لأكاسيد المعادن وبتراكيز مختلفة. وارتفعت قيمة صلابة السطح (shore A) مع زيادة تركيز الجسيمات النانوية في الراتنج المعدل. ان استخدام الجسيمات النانوية وبتراكيز 1 و 2 % من الوزن مضافة الى بطانة المرنة اثرت بتقليل قيمة خشونة السطح. **الاستنتاجات:** اضافة الجسيمات النانوية لأكاسيد المعادن حسنت من ملمس سطح بطانة الطقم المرنة ذات الاساس الاكريلي، في حين أن صلابة سطح البطانة المرنة المعدلة ازدادت مع زيادات تركيز الجسيمات النانوية.

DOI: [10.33899/rdenj.2022.129989.1104](https://doi.org/10.33899/rdenj.2022.129989.1104) , © 2022, College of Dentistry, University of Mosul.

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INTRODUCTION

Tissue side alteration was carried out to ensure the fitness of denture after a period of service due to changing in ridge form can be obtained by replacing an existent inner surface of the denture by relining⁽¹⁾.

The soft liner was used for relining to improve the denture wearers' comfort and improve prosthodontic treatment. The acrylic-based soft liner is a mixture of methyl methacrylate and 30% to 60% plasticizer that mainly responsible for softness and corrupted liner properties⁽²⁾.

Long term uses of soft liners may lead to fungal colonization by *Candida albicans* that enhanced by the presence of saliva and serum pellicles⁽³⁾.

Drawbacks of the relining materials are mainly the lack of a durable bond to the denture base. The liners detachment from the denture base is a common clinical occurrence⁽⁴⁾.

The acrylic-based soft lining seems to be more prone to degradation, where plasticizer loss appears to be due to osmotic gradients, oil solubility⁽⁵⁾.

Discolouration of denture base relining materials is observed after long-term use by accumulation of stain, absorption of water, dissolution of ingredients, microorganism and intrinsic pigments degradation⁽⁶⁾

The addition of silver NPs with different concentration to soft-liner increases the shore A hardness value as the nanoparticles increased,⁽⁷⁾ while, the

addition of TiO₂ nanoparticles to liner displayed hardness values lower than the acceptable range⁽⁸⁾.

Soft liner with silver nanoparticle shows high water absorption and solubility in contrast to low-level concentration of silver.⁽⁹⁾

The addition of silver NPs (0.5, 1, 2, and 3 wt %) and thermocycling of silicon-based soft liner reduces the tensile strength to denture acrylic resin⁽¹⁰⁾.

The addition of silver NPs and ZrO₂ NPs to the soft liner with different concentration shows a significant increase in light absorption and decreased translucency^(11,12).

Fungal colonization on soft liner surface (initial adherence) is influenced by surface roughness, in which rough surface more prone to microorganisms' colonization⁽¹³⁾. The surface roughness was measured by a profilometer⁽¹⁴⁾.

The interest in Magnesium oxide NPs was increased for biomedical applications (such as implants, bone surgery, antimicrobial agents, etc.) due to their biodegradable, nontoxic⁽¹⁵⁾

Zinc oxide (ZnO) is listed as safe by the U.S. Food and Drug Administration that used for a long time as cosmetic or pharmaceutical ointments⁽¹⁶⁾.

ZrO₂ nanomaterials are non-toxic, biocompatible and bioinert so is widely used in medical and orthopaedic applications, mainly replacement of damaged parts of the human skeleton, bones, and teeth⁽¹⁷⁾.

This study aims to evaluate the effect of adding MgO, ZnO and ZrO₂ nanoparticles on surface roughness, shore A, and FTIR of acrylic-based soft liner

MATERIALS AND METHODS

Acrylic-based soft liner (vertex soft, Vertex-Dental, Netherlands) specimens were prepared by adding MgO, ZnO, ZrO₂ (Table 1) nanoparticles at 0.5, 1, 2% Wt concentration to monomer (calculated

from polymer weight and added to monomer to obtain better nanoparticles depression) and sonicated for 5 min by ultrasonic bath to obtain a homogenous distribution of nanoparticles^(18,19).

Soft liner dough was packed and cured in prepared stone mold (with specific dimension for each test) in the water bath for 90 min at 70C° and 30 min at 100 C° (according to manufacturer instruction).

Table (1): Nanoparticles used in This Study

Nanoparticles	Size	Manufacture
Magnesium oxide (MgO)	20-40 nm	Nano shel USA
Zirconium oxide (ZrO ₂)	10-20 nm	Nano shel USA
Zinc oxide (ZnO)	35-40 nm	Nano materials USA

Surface hardness:

Fifty soft liner samples (five for each group) were prepared by investing elastic foils with 30mm diameter and 3mm thickness in dental stone (class IV) to create a mold for acrylic-based soft liner specimens⁽²⁰⁾.

Prepared specimens were stored in distilled water at 37°C for 24 hours before the testing procedure performed⁽²¹⁾.

Shore A durometer (Shore A LX durometer A, Loyka, turkey) was used to measure the hardness of soft liner (study groups and control). The specimens were supported by a metal base of the instrument throughout the testing procedure. The distance between the soft liner specimen and the durometer indenter was fixed at 20mm and 5sec contact time after

penetration. Soft liner specimens hardness were measured by taking the average of three different reading from the device scale⁽²²⁾.

Surface roughness:

Soft liner with nanoparticles samples were prepared by investing elastic foil with 10 mm × 10 mm × 2 mm dimension in dental stone⁽²³⁾. Then the samples were removed from the mold after water bath polymerization. Samples were left intact without smoothing and polishing to represent the surface texture of the denture tissue site, the samples were stored in distilled water for 24 hours at 37C°⁽²⁴⁾.

A profilometer (Talysurf , Taylor Hobson, UK) was used to measure the surface roughness with 0.25µm Dimond stylus head at 0.5mm/s speed and cut off

length 2.5 mm.. The stylus of the device that travels over the whole surface of the measurement of the specimen was recorded (25).

FTIR analysis:

The evaluation of chemical reaction between metal oxide nanoparticles and PMMA of the acrylic-based soft liner was carried out by Fourier Transform Infrared Spectroscopy (FTIR) analysis (PLATINUM ATR, Bruker, Germany) at FTIR spectra in the wavenumber range $4,000\text{ cm}^{-1}$ - 400 cm^{-1} (26).

Sample preparation for FT-IR spectroscopy:

The modified acrylic-based soft liner with metal oxide samples were ground by coarse stone bure (Tokuyama, Japan) to obtain soft liner powder.

Soft liner powder was mixed with Potassium bromide (KBr for spectroscopy) in a ratio (100:2) by Mortar and pestle then the powder was compressed under a pressure of 10 kg/cm^2 for about 30 s to form pellet for FTIR testing (according manufacturer instructions).

Statistical analysis was performed by SPSS statistic software (IBM, USA) to analyze the data of tests. ANOVA and Duncan's multiple range tests were used to evaluate the effect of metal oxide nanoparticles on the acrylic-based soft liner.

RESULTS AND DISCUSSION

Surface hardness:

Table (2) shows that highly significant differences (p -value < 0.05) between the control and tested groups. Duncan's multiple range test (figure 1) demonstrated that, there are no significant differences between the control group and soft liner samples with 0.5% concentration of MgO and ZnO nanoparticles, while there is increased soft liner hardness after modification by nanoparticles (MgO, ZnO, and ZrO_2) at 1 and 2% wt.

Soft liner samples with MgO nanoparticles at 0.5% and ZrO_2 at 0.5% wt concentration show that, the lowest shore A value than the control group with no statically differences.

Table (2): ANOVA of shore A hardness of modified soft liner

	Sum of Squares	df	Mean Square	F	P value
Between Groups	1249.312	9	138.812	36.987	.000
Within Groups	150.119	40	3.753		
Total	1399.431	49			

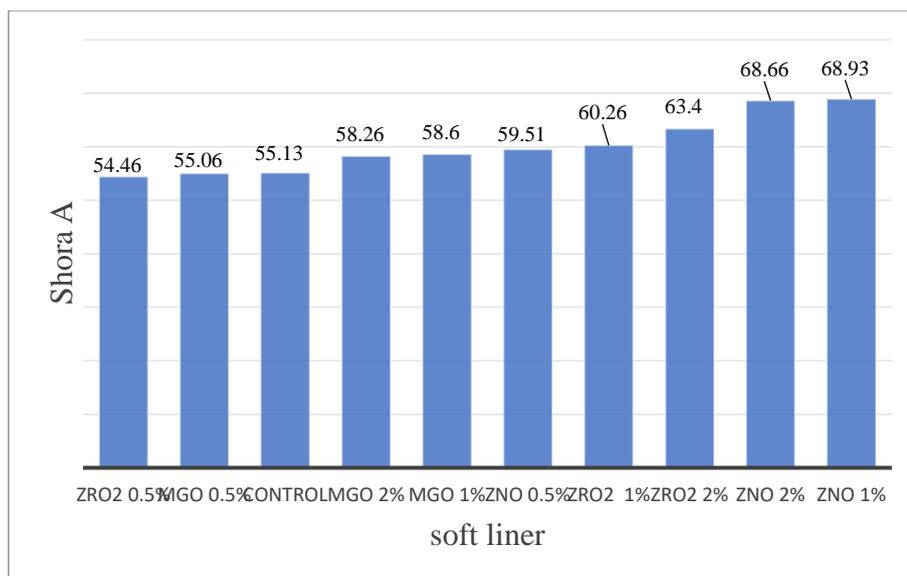


Figure (1): Mean and Duncan's multiple range test of shore A hardness of modified soft liner

Plasticizer content of acrylic-based soft liner controls the softness of the liner, so this will increase in shore A hardness value when a nanoparticles concentration increased, and this may be due to that addition of nanoparticles (as antimicrobial agents) may act as filler that spread inside the gel structure of soft liner, leading to increasing to shore A hardness value ⁽²⁷⁾.

While in lower nanoparticles concentration (0.5 and 1%wt) there is no or low decrease in shore A value, which may be due to nanoparticles act as impurities and increases of unreacted monomer (act as plasticizer), as a result of bad diffusion of metal oxide nanoparticles in PEMA that disturbs degree of conversion ^(28,29).

This result disagrees with Chladek et al ⁽⁷⁾ that may be due to uses of different Nanoparticles types (Ag NPs) and different soft liner materials while agreeing with Urban et al ⁽²⁶⁾

Surface roughness:

The analysis of variance for surface roughness (Ra) of modified soft liner in (table 3) express those significant differences between the control group and tested group.

Duncan's multiple tests of surface roughness (Ra) in μm as shown in (figure 2) reveals that, Soft liner with ZrO₂ at 2% Wt concentration has lower value and significantly statistical differences when compared with other tested groups, while soft liner with MgO 0.5%wt concentration has a higher value of Ra and has no significant statistically differences with control group.

Modified soft liner with ZnO and MgO NPs at 2 and 1% Wt concentration shows decrease surface roughness (Ra) value when compared with control and ZnO and MgO NPs at 0.5%wt concentrations.

Modified soft liner with high metal oxide nanoparticles concentration (1% and 2% wt) shows decreases surface roughness

of modified soft liner in contrast to control group and low nanoparticles concentration (0.5%).

Table (3): ANOVA of surface roughness of modified soft liner

	Sum of Squares	Df	Mean Square	F	P value
Between Groups	1.433	9	.159	30.156	.000
Within Groups	.211	40	.005		
Total	1.644	49			

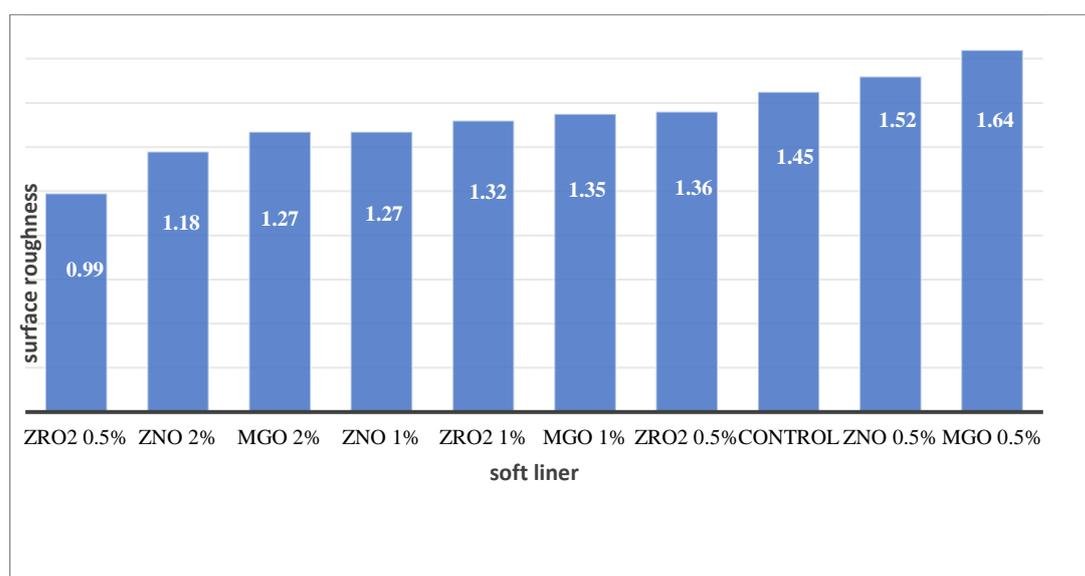


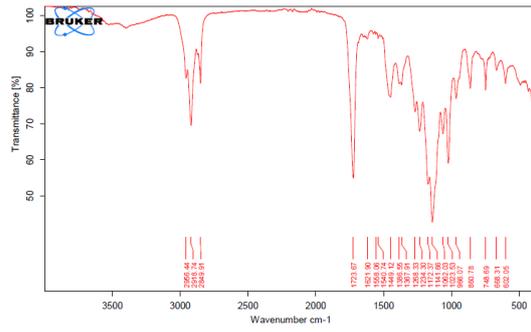
Figure (2): Mean and Duncan's multiple range test of surface roughness (Ra) for a modified soft liner with nanoparticles in μm

This result may be due to the addition of ZrO₂, MgO, and ZnO nanoparticles in high concentration (1 and 2%wt) act as nano filler to fill polymer chains spaces and highly distributed within resin matrix⁽³⁰⁾. While in low concentration the nanofiller may be no enough to fill this matrix. This result disagrees with Jasim *etal.*⁽³¹⁾ who reports that, here are no significant differences of a modified soft liner with alumina nanofiller, which may be due to the

high concentration of uses of different nanoparticles.

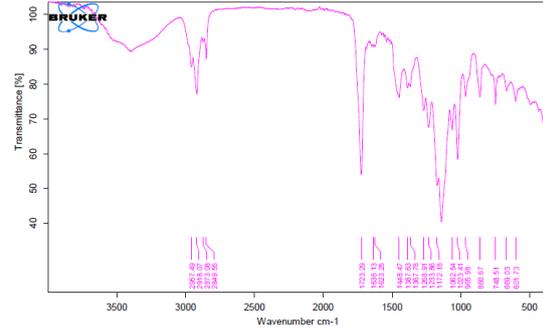
The modified acrylic-based soft liner shows no chemical interaction with ZrO₂, MgO, ZnO nanoparticles at a different concentration than analyzed by the FTIR device as shown in figure (3).

This result may be due to metal oxide nanoparticles used in this study are saturated with oxygen atoms that prevent expected bond nanoparticles and Carbonyl ester⁽³²⁾.



control

This result is agreed with Hasan and Ali ⁽³³⁾ while disagreeing with Atsü and Keskin ⁽³⁴⁾ which may be due to salinization



MgO

of nanoparticles with silane agent that enhances the chemical bond with soft liner.

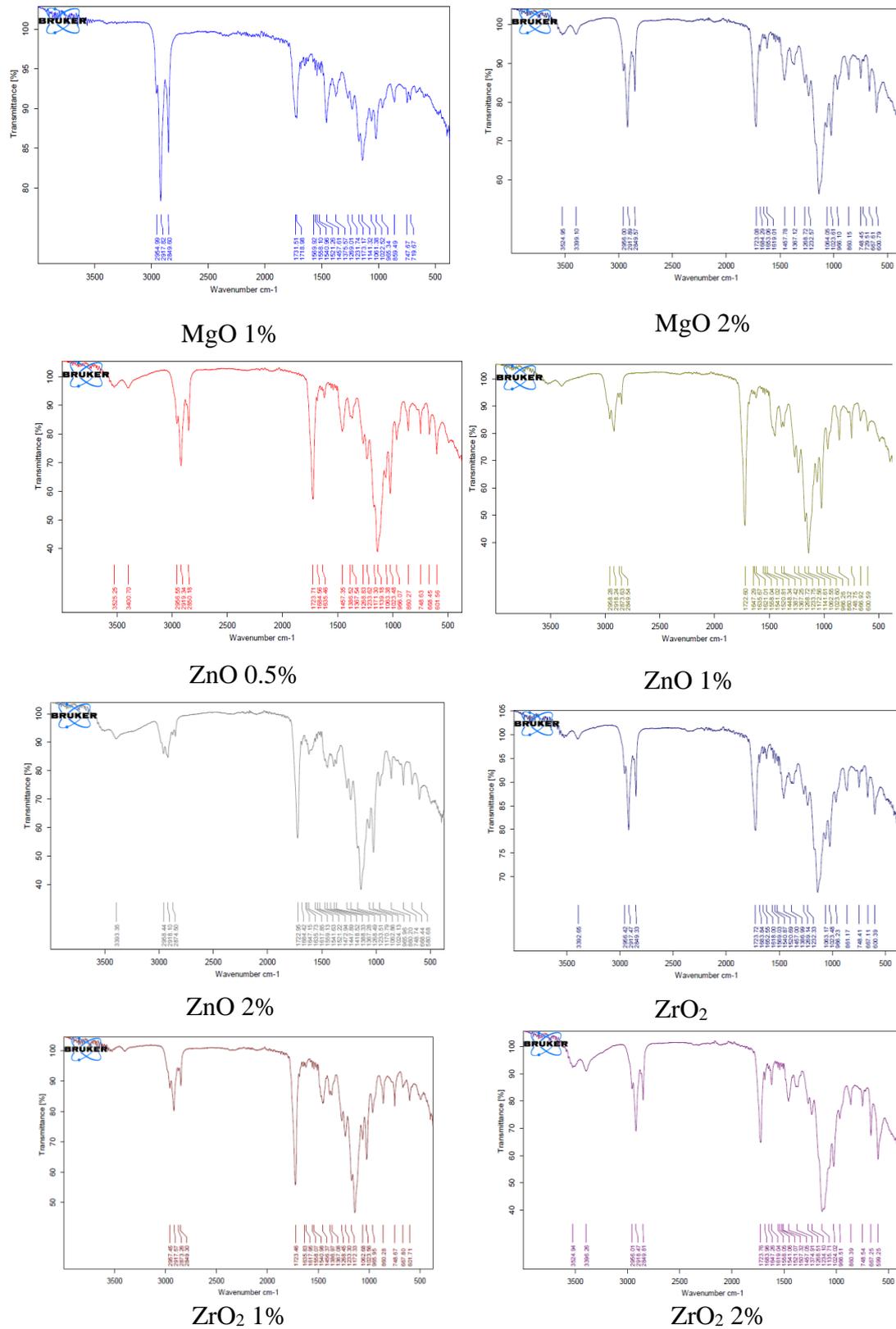


Figure (3): FTIR analysis of modified soft liner with different concentration nanoparticles

CONCLUSIONS

With the limitation of this study, the addition of metal oxide nanoparticles in 2%wt concentration increases surface hardness of modified acrylic-based soft liner, in contrast to low concentration 0.5 and 1% show no effect on surface hardness. Surface texture has enhanced as metal oxide concentration increases in the modified soft liner.

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