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Effect of adding copper oxide nanoparticles on tensile strength and elongation percentage of self-cure soft liners used in prosthodontics

Abstract. Even though polymers are the primary material used in soft liners because of compatibility, chemical inactivity, and easy usage, several properties deteriorate with time and usage, rendering replacement of prostheses unavoidable (every 6—12 months). Multiple studies have been conducted to improve the properties of soft liners, such as tear and tensile strengths, and to retain these properties as long as possible to withstand aging and disinfection. **Aim of study:** Evaluating the influence of adding copper oxide (II) CuO nanoparticles (40 nm) to the soft liner on tensile strength and elongation percentage. **Materials and methods.** Nano-CuO in 1% (wt.) was selected to be added to the self-cured soft liner. A total of 40 specimens were qualified according to the manufacturer and standardization; 20 specimens were tested for the control group, 20 for the modified one, 10 for the tensile strength test, and 10 for the elongation percentage. Fourier transform infrared spectroscopy (FTIR) was used to check whether a chemical interaction would occur between the nano-CuO and the soft liner. **Results.** The average value of tensile strength of soft liner was recorded to be 1.437 MPa before the addition of nano-CuO soft liner, and it was recorded to be 1.808 MPa after enforcement, a significant difference between groups before and after the addition of nano-CuO (p=0.023). The elongation percentage of the liner had a mean value of 461%. The non-significant decrease was documented (p=0.912). The tensile strength was significantly improved, while the elongation percentage was non-significantly decreased. **Conclusion.** Incorporating CuO nanoparticles into the soft liner had increased the tensile strength and decreased the elongation percentage. There was no chemical interaction between the nano powder and the soft liner (only physical dispersion).

Key words: soft liners, copper oxide, nano-powder, reinforcement, tensile strength

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Влияние добавления наночастиц оксида меди на прочность при растяжении и процент удлинения самоотверждаемых мягких подкладок в ортопедической стоматологии

Реферат. Несмотря на то что полимеры являются основным материалом, используемым в мягких вкладышах из-за их совместимости, химической инертности и простоты использования, некоторые свойства ухудшаются со временем и в процессе использования, что делает замену протезов неизбежной каждые 6—12 месяцев. Было проведено множество исследований с целью улучшения свойств мягких вкладышей, таких как прочность на разрыв и растяжение, а также для сохранения этих свойств как можно дольше, чтобы они выдерживали старение и дезинфекцию. **Цель исследования** — оценка влияния добавления наночастиц оксида меди (II) CuO (40 нм) в мягкую подкладку на прочность на разрыв и процент удлинения. **Материалы и методы.** Для добавления в самоотвердевающую мягкую подкладку был выбран нанопорошок CuO в концентрации 1% (масс.). В соответствии с требованиями производителя и стандартами было изготовлено 40 образцов: 20 — для контрольной группы, 20 — для модифицированной. В каждой группе 10 образцов испытывали на прочность на разрыв и 10 — для определения степени удлинения. Для проверки возможного химического взаимодействия нано-CuO и материалом подкладки была использована ИК-спектроскопия с преобразованием Фурье (FTIR). **Результаты.** Среднее значение прочности на разрыв мягкой подкладки до добавления нано-CuO составило 1,437 МПа, а после добавления — 1,808 МПа, что свидетельствует о значительной разнице между группами до и после добавления нано-CuO (p=0,023). Процент удлинения подкладки имел среднее значение 461%. Было зафиксировано незначительное снижение (p=0,912). Прочность на разрыв значительно улучшилась, в то время как процент удлинения незначительно снизился. **Заключение.** Добавление наночастиц CuO в мягкую подкладку повысило прочность на разрыв и снизило процент удлинения. Химического взаимодействия между нанопорошком и мягкой подкладкой не наблюдалось (только физическая дисперсия).

Ключевые слова: мягкие вкладыши, нанооксид меди, армирование, предел прочности

INTRODUCTION

Soft liners used in removable prosthodontics are resurfacing materials added to the tissue surface of dentures for force distribution, improving retention and stability, and resulting in patient acceptance. As a result of continuous residual ridge resorption, dentures often must be relined as part of maintenance follow-up recalls. This will improve tissue contact and occlusal relation with the other arch [1].

Despite their benefits, soft liners suffer aging and lose their cushioning effect, becoming prone to Candida albicans growth, losing their tensile strength, and debonding from the acrylic resin denture base. As a result, techniques are frequently adopted to roughen the acrylic resin denture base by sand blasting, plasma, or etching to enhance the bonding quality [2].

Moreover, research has been done to enhance the properties of soft liners to resist aging. The reinforcement by incorporating nano fillers, such as Yttrium oxide, silicone dioxide, or glass fibers, is a standard reinforcement method [3].

The soft liners may be classified into the plasticized acrylic resin-based and silicone-based resilient lining materials or heat and cold-cure according to polymerization temperature [1].

Additionally, studies have shown that adding certain materials, such as silver and copper nanoparticles, to denture liners can gain antimicrobial effects [4].

MATERIALS AND METHODS

A total of 40 specimens of a silicone-based soft denture liner (Tokuyama, Japan) were made and separated into two equal groups:

1) control group — soft liners without modifications;
2) experimental group — soft liner impregnated with 1% CuO 40 nm nano-particles (Skyspring Nanomaterials, USA).

Specimen manufacturing

CAD software was used to draw and design the dimensions of the mold for specimen manufacturing. Then, a CNC-machine (Computer Numerical Control) was manipulated to form the mold compartment for the soft liner to be poured till setting [5].

For the control group, the mixing gun injected the soft liner through the auto mixing nozzle directly into the mold space. The cover was applied till complete setting of the material after 20 minutes. For the experimental group, the soft liner was injected into the mixing cup on the digital electronic balance (10^{-3} accuracy), then nano-powder was applied at 1%. The soft liner was mixed with the nanopowder using a vacuum mixer for 1 minute at 360 rpm to get the enforced group. A vacuum pressure of −10 bar was applied for 1 min to remove air bubbles. The material was applied to the mold space, and the cover was closed for 20 minutes [3].

The mixture was applied gradually and poured on the acrylic mold excessively to reduce the percentage of air bubbles. The cover was then placed and fixed with screws and nuts. After that, G-clamps were applied on the sides with a constant load application at the top of the cover of 1 kg. The air bubbles that may have formed on the specimen were removed by applying steady pressure until the bubbles had escaped. Then the material was left undisturbed till completely cured [6].

The specimens were kept in standard conditions at 20—25°C and 50—60%. A storage box was designed using a water and heat-proof container with a thermometer and hygrometer. After completing the setting, the specimens were retrieved. The tested specimens must be intact with good borders, and no air bubbles should be present. Defects render the specimen to be discarded. Flashes and excesses were cut out with a scalpel or surgical blade no. 11.

For each of the 20 specimens, 10 were made for tensile strength and 10 for elongation percentage.

Spectrometry

Fourier transform infrared spectrometry (FTIR) was performed on the soft liner before and after adding CuO nanopowder. An identical FTIR would indicate no chemical reaction and only physical dispersion had occurred.

Mechanical testing procedures

ISO 37:2011 (type 2) was implicated. Specimens were prepared in a dumbbell shape and tested by a universal testing machine per the specification. The velocity of 500 mm/min was applied, during which the specimen was pulled until complete rupture. The tensile strength is the highest force at rupture divided by the cross-sectional area. The elongation percentage can be calculated by dividing the change in length by the original length. The specimen length was measured before stretching, and again after rupturing. The relative elongation was then calculated as a percentage.

Statistical analysis

Nominal variables were summarized using frequencies and percentages, while ordinal variables were described using means and standard deviations. A significance level of $p<0.05$ was considered statistically significant. Independent samples t-tests were used to compare means between groups.

RESULTS

Spectrometry

The FTIR spectrum showed the characteristic peaks corresponding to the functional groups present in the soft liner material before and after nano-CuO addition (fig. 1 and 2), and had exhibited similarities in peaks for the soft liner before and after nano-CuO addition as follows:

- peaks at 2963 cm^{-1} indicating the presence of C—H stretching bond for alkanes such as CH_2 or CH_3;
- peaks at 1259 cm^{-1} indicating the C—O bond;
- peaks at 1076 and 1004 cm^{-1} indicating the presence of —O— bond;
- peaks at 863, 786, and 694 cm^{-1} indicating out-of-plane bending of the C—H bond as that of an aromatic compounds (benzene circle).

These results suggest that the compound is alcohol, ester, or R—O—Ar (R = alkane, Ar = vinyl or benzene). The alcohol and the ester are less likely, as no O—H bond or carbonyl group exists. The most likely is the R—O—Ar, which reflects the manufacturer's statement that the silicone-based soft denture liner (Tokuyama, Japan) is composed of divinyl polydimethylsiloxane. The latter further indicates that nano-CuO didn't cause a chemical reaction or chemical structural change (Table 1).

Table 1. FTIR absorption peaks for the soft liner with and without the addition of nano-CuO

Before addition		After addition of nano-CuO	
Wavenumber (cm^{-1})	Absorption (%)	Wavenumber (cm^{-1})	Absorption (%)
2963.0010	16.4	2963.0626	16.6
2907.6418	3.6	2906.4740	3.4
1408.8205	6.5	1408.7270	6.3
1259.1228	51.5	1259.1017	52.0
1076.8340	67.8	1076.9636	67.6
1004.7157	92.9	1004.4584	93.2
863.1010	32.0	863.1093	31.9
786.6476	91.6	786.4429	91.8
694.3476	39.5	694.4137	39.5

Mechanical testing

The average tensile strength of the soft liner was 1.437 MPa before the addition of nano-CuO and increased to 1.808 MPa after reinforcement, indicating an improvement in mechanical performance due to the incorporation of nanoparticles (Table 2). Statistical analysis showed a significant difference among groups before and after adding nano-CuO (p=0.023).

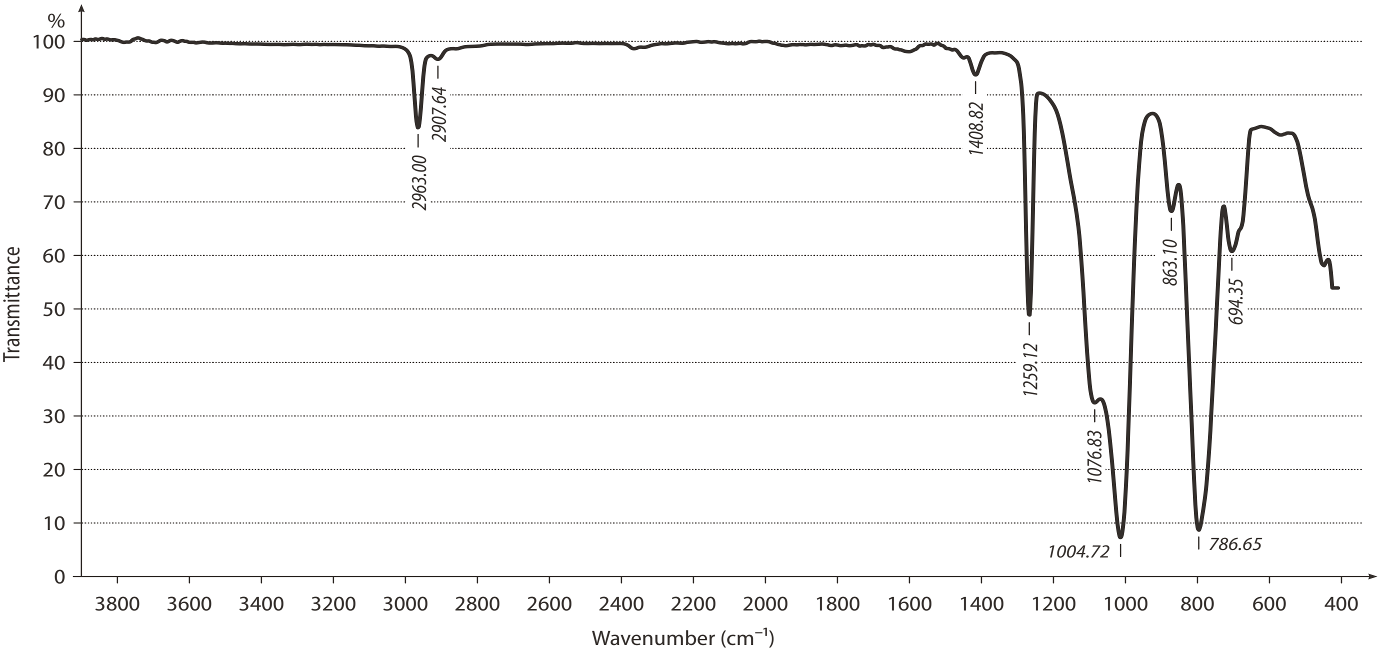

Fig. 1. FTIR of the soft liner before the addition of nano-CuO

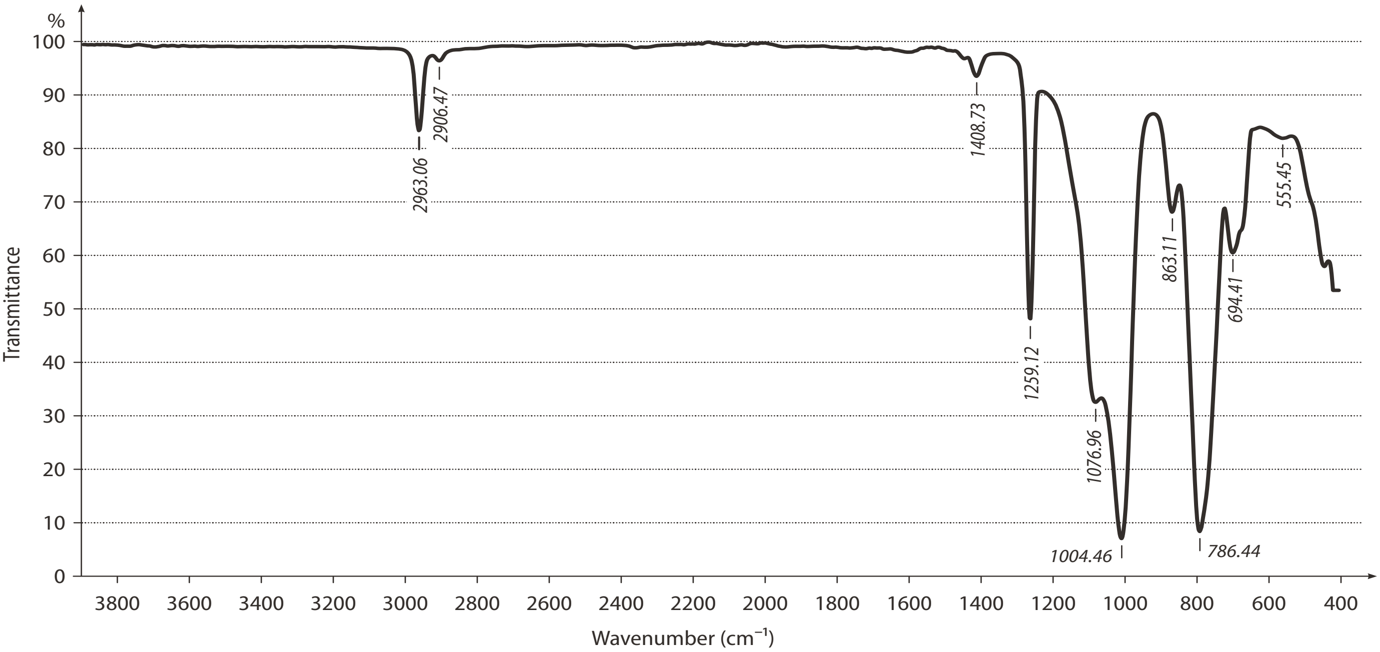

Fig. 2. FTIR of the soft liner after adding nano-CuO

The elongation percentage of the liner had a mean value of 461%, and there was a non-significant decrease in the rate after the addition of nano-CuO, which was recorded at 458%. The non-significant decrease was documented (p=0.912), as shown in Table 2.

When observed under UV light, conventional self-cure soft liners and those modified with nano-CuO show noticeable differences in appearance and stability. The conventional appear smooth uniform translucent with no significant fluorescence. In contrast, the nano-CuO—reinforced liner tend to appear darker (opaque) and scatter. Visual comparison of conventional and nano-CuO modified soft liner under UV light shown in fig. 3.

Table 2. Comparison of tensile strength and elongation of a liner in groups

	Control		Modified by CuO nano-powder		t	p
	Mean	Min—max	Mean	Min—max		
Tensile strength (MPa)	1.437±0.216	1.190–1.730	1.808±0.388	1.350–2.700	2.505	0.023
Elongation (%)	461±50	393–503	458±42	403–504	0.112	0.912

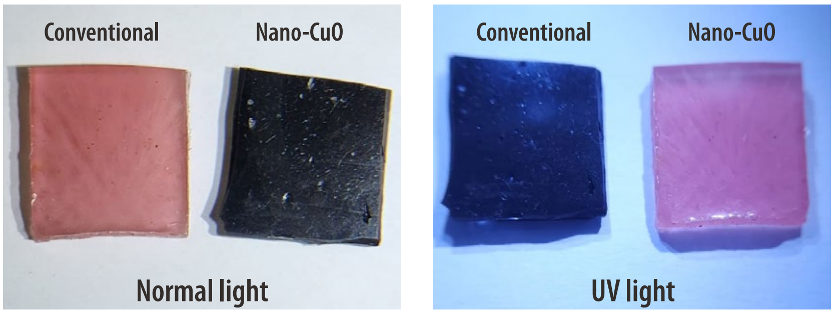

Fig. 3. Visual photography of the comparison of conventional and nano-CuO modified soft liner under normal and UV light

DISCUSSION

Chemically, there is no interaction between nano-powder and soft liner, as there is no change in the spectral range of the soft liner after the addition of the nano-CuO powder, and this can be because the smooth liner is present in the saturated form [6].

The increase may be caused by the fact that nano-CuO powder was physically scattered within the soft liner, so the latter polymer chains with scattered nanoparticles within will slide over each other when subjected to tension due to the applied tensile forces; these chains are more resistant to breakage compared to unmodified soft liners [7].

Because of the ability of nano fillers to form a three-dimensional network with the chains of polymer with subsequent entrapment of these networks within the polymer matrix, the density of cross-linking of the polymer matrix will be increased, causing an increase in stiffness of the material and an increase in tensile strength [8].

The decrease in elongation percentage after the addition of nanoparticles may be explained by restrained molecular chains due to scattered nano powder that disrupts the orientation and flow, causing failure at multiple points and eventually less elongation percentage values compared to unmodified soft liners [9].

CONCLUSION

There is no chemical interaction between CuO nanoparticles and Tokuyama self-cured soft liner (only physical dispersion of the fillers within the silicon matrix) as shown by FTIR. The addition of CuO nanoparticles to the soft liner resulted in an improvement in the tensile strength. The addition of CuO nanoparticles to the soft liner resulted in a decrease in the elongation percentage, although the decline was non-significant.

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