ORIGINAL ARTICLE

Evaluation of Some Properties of Heat Cured Soft Denture Liner Reinforced with Calcium Carbonate Nano-Particles

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ABSTRACT

Aim: To evaluate the effect of adding calcium carbonate nanoparticles ($CaCO_3$NPs) to soft denture lining material on radiopacity, hardness and surface roughness of the lining material. $CaCO_3$NPs with 1.5% and 2% concentration by weight were added into acrylic-based heat cured soft denture lining material.
Methods: Pilot study was carried out to select the best percentage of $CaCO_3$NPs by experimenting their effect on radiopacity, shore A hardness and surface roughness of soft denture liner. Four concentrations of $CaCO_3$NPs were used (0.5%, 1%, 1.5%, and 2%).
Results: Ninety specimens were prepared according to the tests to be formed. The radiopacity of the composite of soft liner/$CaCO_3$NPs was evaluated, shore A hardness and surface roughness were also measured, the results showed that there was a highly significant increase in radiopacity and shore A hardness of the experimental specimens in comparison with the control specimens ,while non-significant difference in the surface roughness was observed in experimental specimens.
Conclusion: The addition of $CaCO_3$NPs can provide soft liner material with improved radiopacity and shore A hardness, while surface roughness was non-significant changed when the concentration of $CaCO_3$NPs increased.
Keywords: Soft denture liner, radiopacity, shores A hardness, surface roughness

INTRODUCTION

Soft denture lining materials are set to denture tissue surface area to provide more equal force distribution, retention and stability of denture, occlusion and patient appearance, and reduced localized pressure[1,2].
The denture should be relined when the underlying residual ridge resorbed and the denture will lose its accurate adaptation on this resorbed ridge, so the soft liner is utilized to improve denture's fitting to the underlying supporting tissues[3].
The resilient lining material can be divided into plasticized acrylic resin or silicone elastomers. The plasticized acrylic resin involves of acrylic polymers and copolymers, a liquid containing an acrylic monomer and plasticizers that is responsible for the protection of the material softness[4,5].

Soft denture liner should have good dimensional stability during and after processing, non-toxic, non-irritant, odorless, stable color, easily processed and used, acceptable esthetic, little water absorption and solubility, sufficient mechanical strength, abrasion and tear resistance, inhabit bacterial and fungal growth and strong bond with denture base material to avoid de-attachment during use[5-8].

A number of problem usually are associated with use of resilient denture liner, including failure of the bond between soft lining material and denture base, loss resiliency, surface roughness and porosity, poor tear strength and colonization by candida albicans[9,10].

One of the important problem of the soft denture liner is radiolucent and cannot be detected by using normal radiographic technique, therefore if patient swallow or traumatically impact the removable denture, there will be difficult in identifying these objects radiographically and in some cases this requires the utilize of advanced radiographic technique. Delay in identifying and removing such foreign object may threaten the patient's life[11].

Several studies had been done to enhancement properties of soft denture liner by addition different types of filler. Inorganic nanoparticles drastically improve the physical and mechanical properties of soft denture liner even though the amount of addition is less[12-14].

To improve the properties of polymer nanocomposites that result from incorporation inorganic nanoparticles are depend on the type of incorporating nanoparticles, their shape and size,also their concentration and interaction with the polymer matrix[15].

Calcium carbonate nanoparticles ($CaCO_3$NPs) is most common, biocompatible, available and inexpensive inorganic filler that used in nanocompsite process. Nanoparticles of Calcium carbonate has potential to be an important functional filler in polymer systems such as polyproplene composite and polyvinyl chloride composite. Using $CaCO_3$NPs improved the mechanical properties and radiopacity of polymer[16-19].

The current study was conducted to evaluate the effect of incorporation of $CaCO_3$ nanoparticles into heat cured soft denture liner material at different concentration in attempt to improve radiopacity and some other properties.

MATERIAL AND METHOD

Pilot study was carried out to select the best percentage of $CaCO_3$NPs by experimenting their effect on radiopacity, shore A hardness and surface roughness of soft denture liner. Four concentrations of $CaCO_3$NPs were used (0.5%, 1%, 1.5%, and 2%).

According to result of pilot study, 1.5% and 2% of $CaCo_3$NPs were the most appropriate concentrations showed favorable improvement in radiopacity of the soft

liner. Therefore; the main study was conducted by addition of $CaCO_3$NPs with concentration of 1.5% and 2% by weight into polyethylene methacrylate soft denture liner (PEMA) (Vertex™ Soft, Vertex-Dental, Netherlands). Ninety specimens were prepared and divided into three groups according to the tests to be formed, each group was then subdivided into three subgroups according to weight percentage of $CaCO_3$NPs which was added, (control group without $CaCO_3$NPs, experimental groups with 1.5% and 2% $CaCo_3$NPs). For each percentage 10 specimens were fabricated, totally 30 specimens for each test.

Scanning electron microscope (AIS2300C, Angstrom Advanced Inc., USA) was taken for both control (PEMA) and experimental containing 1.5% and 2%$CaCo_3$NPs) specimens to show the degree of $CaCO_3$NPs dispersion within the PEMA matrix. As well as energy dispersive X-ray spectroscopy(EDS) used in conjunction with scanning electron microscopy to show the percentages of composite filler of both weight and atoms. Fourier transform infrared spectroscopy (FTIR) analysis (Tensor 27, Bruker, Germany) was conducted to investigate the chemical interaction between $CaCo_3$NPs and PEMA.

Radiopacity test: Thirty soft liner specimens with dimensions of 2.5 mm in thickness, 10mm in width and 30mm in length were prepared for radiopacity tests [19]. In order to make the dough with particular concentration of nanofiller, the measured amount of nano $CaCO_3$ was added to the monomer and the nanoparticles are distributed well within the monomer with the use of probe sonication apparatus (120 W, 60 KHz) for 3 min. to distribute them into individual nanoparticle[15, 20]. The powder of the soft lining material was added with proportion of (1.2 g of powder/1ml of liquid), then mixed, packed and cured according to manufacturer's instructions.

After complete curing, all the specimens were finished, polished and immersed in distilled water and kept in the incubator at 37°C for (48) hours before testing.

Aluminum plate was cut into the specified shape and measurements, the step wedge comprises of ten stepper beginning from 1mm of aluminum thickness and reaching to 10mm in thickness with 1mm increases at each step[21]. Thirty specimens (ten for each concentration) were arranged over a wax plate of 10mm thickness to simulate the media of soft tissue absorption and reflection. The aluminum step wedge was fixed beside the specimens for standardization of the density of the radiographic film[22].

The specimens, aluminum step wedge and wax plate were placed over cassette (kodak medical x-ray film) and irradiate with 50 Kv, 200 mA and exposure time 0.1 sec. with 1m distance between the source of x-ray and specimens. The computed radiographic system (CR) was used as source of x-ray (Chest X-ray machine, Germany).

The processing was done according the manufacturer's instructions by using automatic x-ray film processor (JP-33, Korea). Light transmission densitometer was utilized to measure the optical density of each step of aluminum wedge and each soft liner specimens. Three readings were taken from each specimens, after that the mean value of these data was calculated[19].

Shore A hardness test: Thirty specimens with dimensions of 3mm in thickness and 30mm in diameter were prepared to be used for shore A hardness test. All specimens immersed in distilled water and kept in the incubator at 37°C for (48) hours before testing. Shore A durometer (Time group-TH200, China) was used to measure hardness of the soft liner samples [23]. Five reading from the durometer were taken for each sample and the contact time was 5 second after each penetration, its average considered the value of the test.

Surface roughness test: Thirty specimens with dimension of (65mm x 10 mm x 2.5 mm) were prepared to be used for surface roughness test. All specimens were immersed in distilled water at 37°C for 48 hours before being tested. The profilometer device (Time3200/TR200, China) was used to study the effect of $CaCO_3$NPs reinforcement into soft liner on the surface roughness of the specimens, three measurements were done for each sample and the average value was calculate [15, 24].

Statistical analysis: SPSS (statistical package for social science) computer software (version 21) was used for analyzing the results of the study, descriptive statistic and inferential statistics were employed. One-way ANOVA (analysis of variance)test was performed, "P" value of > 0.05 was considered statically a non-significant, d" 0.05 was considered a significant and d" 0.01 was considered as a highly significant.

RESULTS

FTIR Analysis: FTIR results showed that there was no chemical reaction between acrylic soft-liner powder and $CaCO_3$NPs powder as shown in (Figure1).

SEM Examination: The morphology of samples in cross section and mapping of samples with PEMA soft liner before and after addition of $CaCO_3$NPs are investigated (Figure 2).

Energy-Dispersive Spectroscopy(EDS): It signalized the presence of ratio of elements as Calcium, Carbon and Oxygen in the liners before and after addition of 1.5% and 2% of $CaCO_3$NPs as shown in (figure 3, 4, 5).

The results of optical density of control group exhibited the highest mean value of 1.636, followed by specimens containing 1.5% wt. CaCo3NPs with 1.533, while lowest mean value of 1.423 was obtained in specimens containing 2% wt. $CaCO_3$NPs.

One way ANOVA test for optical density results showed a highly significant difference among all tested groups as shown in table (1),while to compare the significance of difference between two independent means, Post-hoc Tukey Honestly Significant Difference (Tukey HSD) test was used as in table (2), it revealed that there was a highly significant increase among all tested groups.

Shore A hardness test: Shore A hardness of specimens containing 2% wt. $CaCO_3$NPs exhibited the highest mean value of 57.680, followed by specimens containing 1.5% wt. $CaCO_3$NPs with 53.120, while lowest mean value of 46.220 was obtained in control group. One way ANOVA test for shore A hardness results showed a statistically highly significant difference among all studied groups as shown in table (3), while to compare the significance of difference between two independent means, Post-hoc Tukey Honestly Significant Difference (Tukey HSD) test was used as in table (2), it revealed that there was a highly significant increase among all tested groups.

Surface roughness test: The result of surface roughness test of specimens containing 2% wt. $CaCO_3$NPs exhibited the mean value of 1.3696 µm nearly the same for specimens containing 1.5% wt. $CaCO_3$NPs with 1.3689 µm and for control group with 1.3687 µm. One way ANOVA test for surface roughness results showed a non-significant difference among all tested groups as shown in table (4).

Figure 1: FTIR of (A)soft liner without addition ,(B) soft liner with addition of 1.5% $CaCo_3$NPs and (C) soft liner with addition of 2% $CaCO_3$NPs

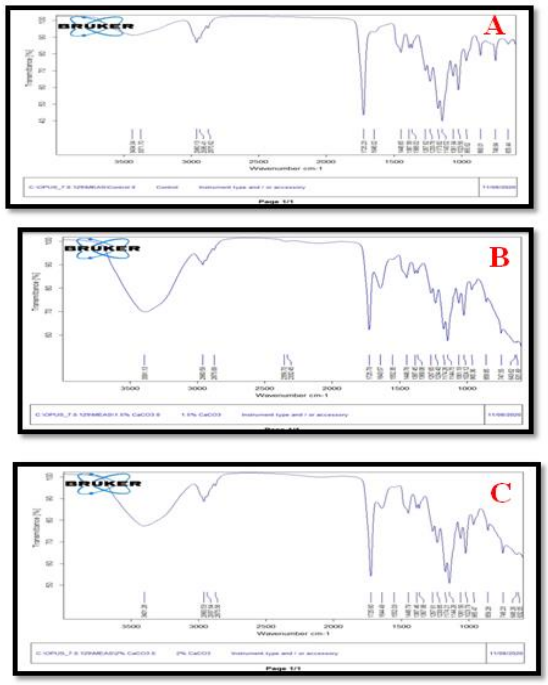

Figure 2: (A) SEM image of control specimen at100um, (B) SEM image of experimental specimen (1.5% $CaCO_3$NPs-PEMA) at 100um and (C) SEM image of experimental specimen (2% $CaCO_3$NPs -PEMA) at 300um.

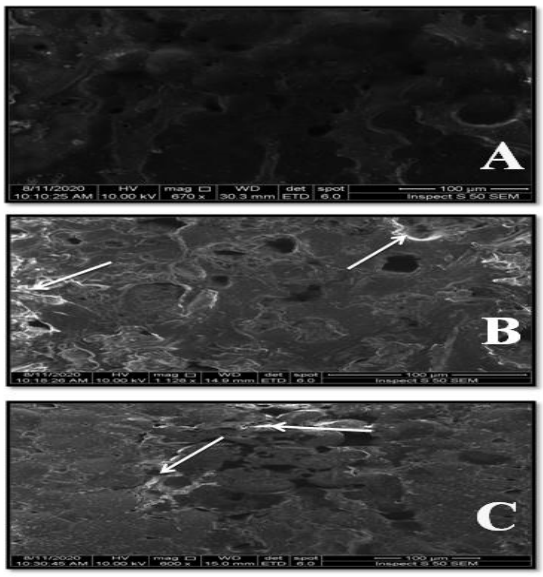

Figure 3: EDS diagram before the addition of $CaCO_3$ nanoparticles

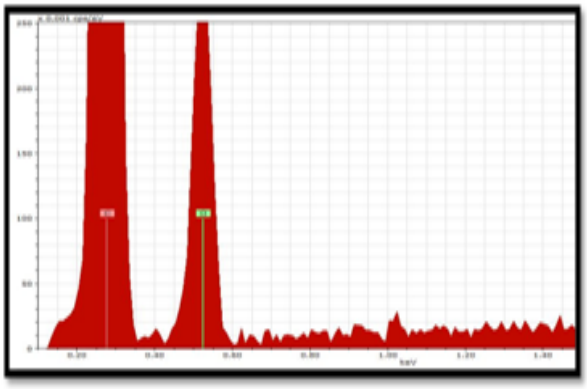

Figure 4: EDS diagram after the addition of 1.5% $CaCO_3$ nanoparticles

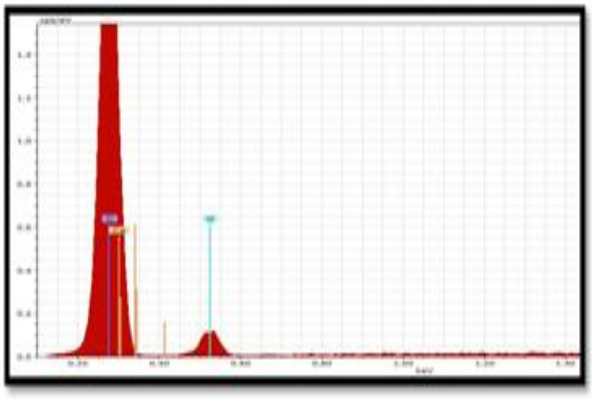

Figure 5: EDS diagram after the addition of 2% $CaCO_3$ nanoparticles radiopacity test

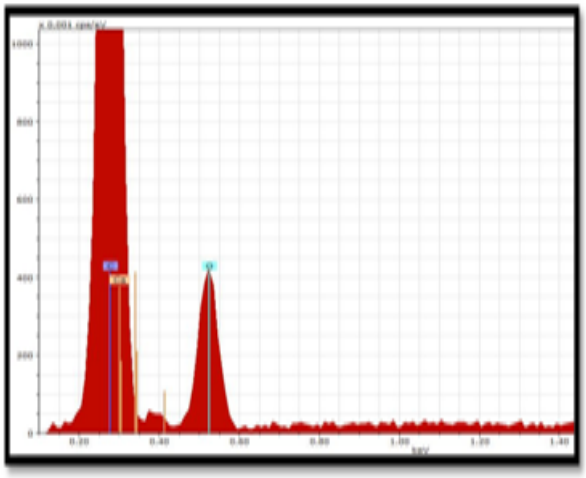

Table1: Means of optical density and ANOVA test for all studied groups.

Groups	N	Mean	SD	SE	Minimum	Maximum	F	P value
Control	10	1.6360	.0303	.0096	1.5900	1.6800	162.434	0.000 HS
1.5% $CaCO_3$nps	10	1.5330	.0295	.0093	1.5000	1.5800		
2% $CaCO_3$nps	10	1.4230	.0177	.0056	1.4000	1.4600		

Table2: Multiple comparisons of optical density and shore A hardness between percentages using Tukey HSD

Variables	(I) Groups	(J) Groups	Mean Difference (I-J)	P value
Optical Density	Control	1.5%CaCO3nps	.1030	.0000 HS
		2%CaCO3nps	.2130	.0000 HS
	1.5%CaCO3nps	2%CaCO3nps	.1100	.0000 HS
Shore A hardness	Control	1.5%CaCO3nps	-6.9000	.0000 HS
		2%CaCO3nps	-11.4600	.0000 HS
	1.5%CaCO3nps	2%CaCO3nps	-4.5600	.0000 HS

Table 3: Means of shore A hardness and ANOVA test for all studied groups.

Groups	N	Mean	±SD	±SE	Minimum	Maximum	F	P value
Control	10	46.220	1.627	.514	43.500	48.300	126.223	0.000 HS
1.5%CaCO3nps	10	53.120	1.454	.460	49.600	55.300		
2%CaCO3nps	10	57.680	1.776	.562	53.700	59.600		

Table 4: Means of surface roughness and ANOVA test for all studied groups.

Groups	N	Mean	±SD	±SE	Minimum	Maximum	F	P value
Control	10	1.3687	.0851	.0269	1.1950	1.4710	0.000235	0.999 NS
1.5%CaCO3nps	10	1.3689	.0881	.0279	1.1840	1.4730		
2%CaCO3nps	10	1.3696	.1162	.0367	1.1390	1.4860		

DISCUSSION

In the present study $CaCO_3$ nanoparticles had been selected as reinforcing nanofillers because it is white in color that is not expected to adversely affect the esthetic appearance of the acrylic denture base, also acted as an excellent reinforcing agent in other polymers like polypropylene, as well as due to its excellent biocompatibility[25].

There are cases where broken pieces of removable denture being ingested or swallowed accidentally, so it is very important for removable denture material to have appropriate radiopacity. As this provides a quicker observation of these pieces before they threaten the patient's life, therefore development of radio paque denture material is a subject of great importance to the researchers[26].

In the current study an extra oral radiograph used since it was the commonly used as a diagnostic radiograph in case of emergency[27].

In radiopacity test, an aluminum step wedge used to standardize the densities of exposed films as aluminum has a coefficient of linear absorption similar to structure of human being like enamel. In addition to the ISO standards which accept radiopacity of 2mm of aluminum thickness. At the same time the use of the base plate wax with 10mm thickness so as to duplicate the scattering and absorbing media of soft tissue[21].

The results of adding calcium carbonate nanoparticles showed that there was a statistically highly significant increase in radiopacity as compared with control group. When the concentration of calcium carbonate nanoparticles increase, there was a statistically highly significant increase in radiopacity but still not reached the ISO standards, this finding can be explained that calcium has lower atomic number compared to other nanofillers like Zirconium and Titanium which produce more radiopacities when incorporated to polymer [19, 28].

In this study, adding 2% of calcium carbonate nanoparticles to soft denture liner material resulted in statistically highly significant increase in radiopacity but still not radio paque enough according to ISO.

This study agree with Mikeal et al in 2018 found that addition of calcium carbonate nanoparticles resulted in highly significant increase in radiopacity of acrylic resin denture base material [19]. The hardness of soft denture liner is an importance property or the softness of them. Soft liner with high level of hardness is undesirable, because it will be less ability to absorb impact effect, for that it's incline toward to utilize a low hardness materials [29, 30].

The results of the study showed that significant increase in hardness with addition of calcium carbonate nanoparticles compared with control group. The increase in hardness is direct proportional with increase in calcium carbonate nanoparticles content. The elastic property that resulted from inter-molecular force or inter-atomic of the material, has the responsibility for the property of elasticity. The stronger attraction forces the more values of the elastic modulus, the more rigid or stiffness material[31].

The soft liner reinforced with $CaCO_3$NPs showed higher surface hardness because with addition of $CaCO_3$NPs the distance between the particles inside the matrix will be decrease. This decrease in the inter-particles distance lead to increase the bonding strength between the particles and cluster together which result in surface accumulation of hard material particles $CaCO_3$NPs in soft liner matrix spaces which result in improved hardness[32].

The increase in hardness is concentration dependent that means increase was slight with low concentration of nanoparticles (as in 1.5% wt. nanoparticles) due to low network density. While highly increase in hardness of the nanocomposite with higher concentration (as in 2% wt. nanoparticles) was attributed to increase in the accumulation of the particles of hard material into the soft liner matrix[33].

In this study, the increase in hardness values of $CaCO_3$NPs incorporated specimens may be attributed to presence of rigid fillers in the soft lining material which effecting restrict surface indentation, this result agree with previous study by Kamil and Al-Judy, 2019 who stated an increase in hardness of PMMA nanoparticles after addition silicon carbide nanoparticles in the acrylic resin[34].

Surface roughness is an important property for soft liner materials because it impacts the adhesion of microorganism and advancement of pathologies such as denture stomatitis, in this way to avoid this formation and to improve oral hygiene, these materials should be in a perfect world show with smooth surface[35].

The roughness of acrylic resin is primarily affected by material inherent feature, operator's skills and polishing procedure so the roughness values are diverse among the other studies because of methodology used, disparities within the experimental procedures as well as the type of acrylic resin and measuring the surface roughness procedure[36].

The results of this study showed that surface roughness of soft denture liner which was not significantly increased after the addition of $CaCO_3$NPs. When small percentages of calcium carbonate nanoparticles were added to soft denture liner only few particles will be included with the surface of the specimen which didn't influence in surface roughness as the surface roughness test is concerned with outer surface and not with the inner surface of composite, too since $CaCO_3$NPs have very small sized particles and well dispersion.

CONCLUSIONS

The incorporation of $CaCO_3$NPs powder into acrylic soft lining material results in a statistically highly significant improvement of radiopacity and shore A hardness, while a non-significant effect on the surface roughness was observed.

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