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Effect of Silver and Zinc Oxide Nanoparticle Addition on Microhardness and Depth of Cure of Resin Based Pit and Fissure Sealants

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ABSTRACT

A previous Iraqi study demonstrated that the addition of silver and zinc oxide nanoparticles resulted in a significant antibacterial action against cariogenic bacteria, when mixed with both fluoridated and non fluoridated sealants.

Aims of the present study: Tend to evaluate hardness and depth of cure of the sealants after the addition of silver and zinc oxide nanoparticles.

Materials and Methods: 120 disk shaped specimens of the sealants were prepared, after polymerization , 60 disks were mounted with their top surfaces exposed and 60 with their bottom surfaces exposed and they were tested for hardness and depth of cure after adding 3 and 7 % w/w addition of silver and zinc oxide nanopoarticles for both sealants.

Results: Showed that the non-fluoridated sealant was harder than the fluoridated, with a statistically significant increase in microhardness after the addition of silver and zinc oxide nanoparticles for both sealants, sealants mixed with silver being the hardest for both groups, sealants before and after the addition had harder top surfaces compared with bottom surfaces, hardness ratio varied between 0.92-0.81.

Conclusion: Within the limits of this study, the addition of silver and zinc oxide nanoprticles increased the microhardness for the fluoridated and non fluoridated sealants, which made them more resistant to wear.

Keywords: Fissure Sealant, Caries, Nanoparticles, Microhardness.

INTRODUCTION

In order to prevent dental caries, several methods have been used including dietary control to restrict the intake of cariogenic food and induce the intake of non cariogenic food, tooth brushing instruction to effectively remove dental plaque, systemic and topical use of fluoride and use of pit and fissure sealants to protect the tooth areas most susceptible to caries [(1-5)]. The occlusal surfaces of the newly erupted posterior teeth are particularly susceptible to carious lesions due to local conditions such as incomplete maturation of the enamel, infra-occlusion and very complex occlusal anatomy. Removing the bacterial plaque under such conditions is difficult, and those surfaces are, consequently, the most affected by caries[(6)]. Fissure sealants have been recognized as an effective approach in preventing pit and fissure caries in children[(7)], when used carefully according to the recommendations. A previous Iraqi study evaluated the antibacterial effect of different nanoparticle addition to a fluoridated and non fluoridated pit and fissure sealant. Silver and zinc oxide nanoparticles when mixed at 3 and 7 % w/w respectively, proved to be very important as antibacterials when mixed with both types of sealants against Streptococcus mutans [(8)] producing zones of inhibition of more than 13 mm. The current study aims to study the microhardness and depth of cure of the fluoridated and non fluoridated pit and fissure sealants after the addition. According to the ANSI/ADA Specification No. 39, ISO 6874[(9)] for Polymer-based pit and fissure sealants, the depth of cure for light activated sealants should not be less than 1.5 mm. If the material is supplied in more than one shade, each shade, should comply with this requirement.

MATERIALS AND METHODS

Two fissure sealants were used in the study a non fluoridated fissure sealant Natural Elegance pit and fissure sealant Henry Schein (Germany) and a fluoridated sealant Conseal F pit and fissure sealant Southern Dental Industry (Australia). The technical profile and composition of each is presented in Table (1).

Table (1): Technical Profile of the Sealants

Fissure Sealant Company	Composition
Conseal f (Southern Dental Industry)	**Acrylic monomer** **Silica** **Titanium dioxide** **Sodium fluoride** **Including 7% filler particles filled with a submicron filler size of 0.04 microns**
Natural Elegance (Henry Schein)	**Diluted BIS-GMA based resin** **White opaquer** **Microfine silica particles** **Photosensitive catalyst system**

Commercial nanoparticles of silver and zinc oxide were procured from Alfa Aesar Company (Germany), the characteristics and features of the nanoparticles are presented in Table (2).

Table (2): Characteristic Features of the Nanoparticles

Formula	Form	Purity	Particle size (nm)	LoT
Ag	**Powder**	**99.9%**	**20-40**	**C09Y011**
ZnO	**Powder**	**99%**	**20-30**	**I07W013**

The nanoparticles were weighted with the use of a sensitive three digit electrical balance (Kern & Sohn, Germany) on a sterilized mixing pad and the sealant was weighted in to a plastic container tube, the nanoparticles were added in to the container with the sealant and mixed with a plastic spatula and then subjected to five minutes of ultrasonication by a special ultrasonic device(JGC Co., Gremany) to ensure uniform distribution of the nanoparticles in the sealants[(10)]. For both the fluoridated and non fluoridated sealants, silver and zinc oxide nanoparticles were added at 3 and 7 % w/w addition respectively, 120 disk shaped specimens of the sealants were fabricated by the use of special polyethylene molds 5mm in diameter and 2mm in height ,that were placed over a glass slide, after the sealant placement in the molds a glass slide was placed over the top of the material, the samples were polymerized for 40 seconds with LED light from the top surface only, the LED curing output was checked and found to be 804 mW/cm^2 (Coxo Medical, China), after curing the cover glasses were removed and the plastic molds were separated, the samples were than mounted in special stone blocks, 60 samples were mounted with the top surface exposed, the other 60 samples were mounted with their bottom surfaces exposed Figure (1),with a total of 10 samples in each group. A Vickers microhardness tester (OTTO WOLPERT- WERKE ,Germany) was used to perform specimen indentation Figure (2). Measurment of surface hardness for both top and bottom of each sample was performed for each sealant and after nanoparticle addition , a 500 gm load was applied with a dwell time of 10 seconds. Measurment was performed 3 times for each surface of the sample[(11)] Figure (3). For a given specimen, the three hardness values for each surface were averaged and reported as a single value at each time. Ten readings were obtained for each material on top and bottom surfaces. The diagonal length impressions , d_1 and d_2 Figure(4), were measured under the microscope using a graduated ruler, then the measurement obtained was divided by the magnification power and the average was calculated according to the following formula :

$$d = (d1 + d2) /2$$

Then Vickers hardness number was calculated according to the standard formula[(12)] :

$$VHN = 1.853\ p / d^2$$

Where p is the indentor load , and d is the diagonal length impression.
The hardness ratio was obtained by dividing the bottom surface hardness value by that of the top. Hardness ratio of the specimens were calculated and tabulated using the formula:
Hardness ratio = VHN of bottom surface / VHN of top surface. [(13)]

STATISTICAL ANALYSIS

Statistical analysis was performed using SPSS programme version 16 means and standard deviation were calculated, Student T test was used to test significance between the two types of sealants, and differences between top and bottom surfaces, Duncan's Multiple Range test was used to test significance between the sealants after the addition of the nanoparticles for both sealants. Results were considered significant when $P \leq 0.05$.

Figure (1): Orientation of the Groups

Figure (2): Vickers Microhardness Tester

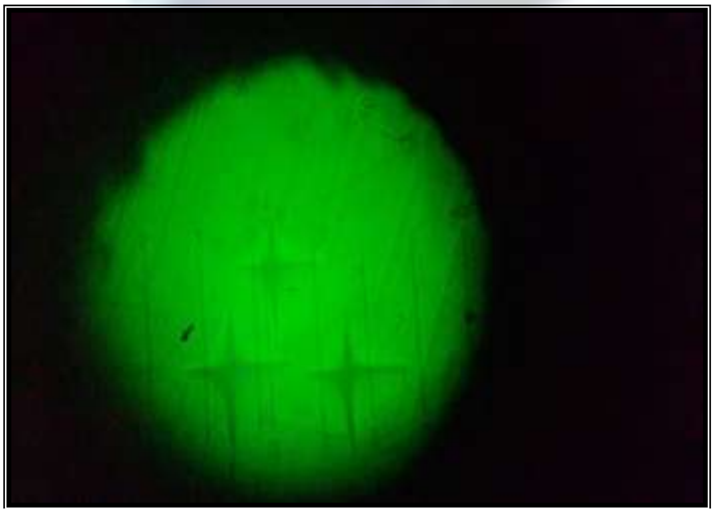

Figure (3): Three Indentations Made on Each Surface of the Sealant

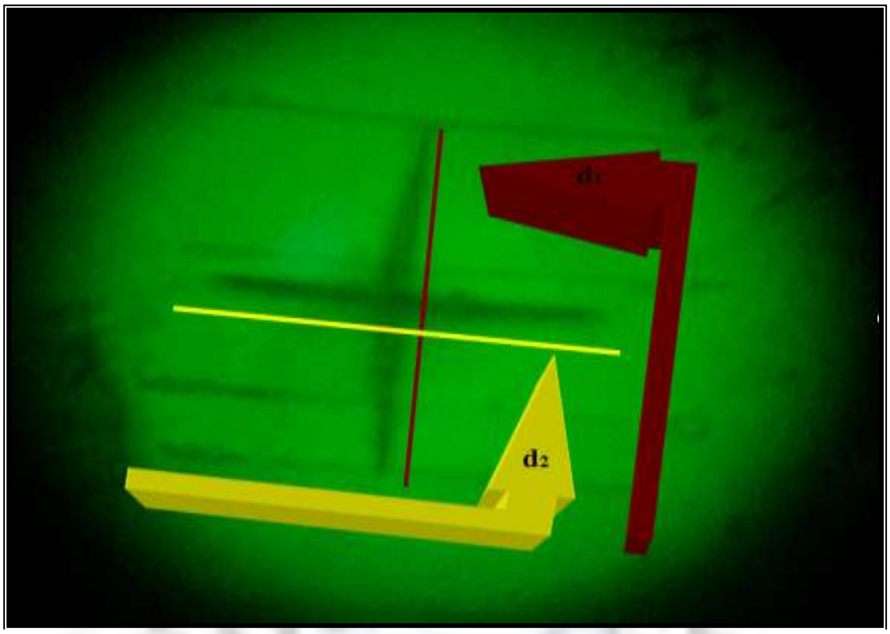

Figure (4): Method of Calculation of Diagonal Length

RESULTS

Table (3) displays the initial microhardness values of both sealants, the non fluoridated sealant had a higher microhardness value than the fluoridated fissure sealant with a statistically significant difference. Table (4) shows the mean microhardness scores for both sealants, and the results after the addition of 3 and 7% w/w nanoparticles of silver and zinc oxide nanoparticle. For both, there was an increase in mean microhardness scores after the addition. It can be depicted from Table(5) that there was a statistically significant difference in mean microhardness of the sealants after the addition, the sealants that were mixed with 3% silver nanoparticles had the highest microhardness score, followed by the sealants that were mixed with zinc oxide nanoparticles compared with the fissure sealants before the addition, for the fluoridated and non fluoridated types of sealant respectively. Table (6)demonstrates mean microhardness values for the two sealants before and after the addition of the nanoparticles for top and bottom surfaces, in general mean microhardness scores for the sealants was greater for top surfaces when compared to bottom microhardness values with statistically significant values. Table (7) displays the hardness ratio of the sealants before and after the addition of the nanoparticles, hardness ratio ranged between 0.92 and 0.81,with the non fluoridated type of fissure sealant showing higher ratios for bottom to top surfaces when compared with the fluoridated one.

DISCUSSION

The use of pit and fissure sealant is an essential and important form of prevention, clinical success of fissure sealants is well-documented in the literature and directly related to its capacity to remain bonded to occlusal pits and fissures. Sealants after polymerization form a strong micromechanical bond to the etched enamel surface, by physically obturating susceptible areas of the tooth surface and preventing dental caries(5). Hardness, on the other hand, can be defined as the resistance of a material to indentation or penetration. Measurement of surface hardness is an indirect method of evaluating the polymerization degree of composite resin(13). Hardness tests are simple and relatively inexpensive when compared with other tests that evaluate the mechanical properties of materials. Hardness tests are used more frequently than any other mechanical test to evaluate material, however, it is difficult to formulate a definition that is completely acceptable for hardness, as the indentation produced results from the interaction of numerous properties among the properties related to hardness of a material such as strength, proportional limit and the ability of the sealant to abrade or by abraded by opposing dental structures and materials (14). Vicker's hardness test is easy to apply and the data obtained are reliable. The diamond indenter used in the procedure does not deform over time and is reportedly suitable for measurement of the hardness of fragile materials and dental tissue (15). According to Kim et al (2002) (16) "material loss in the pits and fissures is thought to be the key factor in the caries preventive function". Early loss is mainly related to moisture contamination during fissure sealing and improper polymerization. The ongoing sealant loss is due to masticatory wear, shear strength weakness, thermal changes and marginal leakage.

In general the microhardness values of pit and fissure sealants are much less than that reported for other types of composite resins(13,17), this might be attributed to the reduced filler particles present in the fissure sealant material which makes it viscous and capable of flowing in to pits and fissures to obliterate them compared to composite restorations .

Initially, the non fluoridated sealant exhibited larger microhardeness values compared with the fluoridated one with a statistically significant difference, this might be attributed to the reason that the filler particle present in the fluoridated sealant according to the manufacturer was not more than 7% including the fluoride particles incorporated in it ,the filler particles in the non fluoridated sealant was not recorded in the sealant pamphlet. By incorporating fillers into both types of the resin-based sealants, an improvement of the hardness values was observed, incorporatation of 3% w/w silver nanoparticles and 7% w/w zinc oxide nanoparticles resulted in increased microhardness of both sealants , with fissure sealants incorporating silver being the hardest, then sealants mixed with zinc oxide with a statistically significant difference, this might be attributed to the reason that silver is a metal and zinc oxide is a metallic oxide and metals are harder than metal oxides even at lower concentrations, an increase in the microhardnees will have advantage that the sealants will be more resistant to wear.

The wear rate of a great number of restorative materials has been studied, however , there are few studies about wear of different materials used as fissure sealant. This property is important since fissure sealants is an extra coronal addition to the occlusal surface and its wear characteristics might be a little different from those that are found in restorative materials that are placed in cavities as fillings (18). Microhardness values for the top surfaces was higher than the bottom surfaces for both sealants, in all the groups, with a statistically significant difference, this might be due to the reduced energy reaching the lower layers of the sealant , thus affecting the final hardness on the bottom surface of all the sealants. This is in agreement with other types of composite resins(13,17,19) . The top and bottom surface hardness values gathered from in vitro studies do not always indicate inadequate polymerization. To define depth of cure based on top and bottom hardness measurements, it is common to calculate the ratio of bottom/top hardness, and give an arbitrary minimum value for this ratio. In order to consider the bottom surface as adequately cured, values of more than 0.80 have often been used (20,21) .

The hardness ratio in the current study varied between (0.92 and 0.81), with the fissure sealants mixed with silver nanoparticles having less hardness ratio compared with the other groups. But it was higher than the minimum value indicated in literature in order to consider the bottom surface as adequately cured (0.80). The depth of cure is dependent on different cofactors such as filler particle size and distribution, colour and optical translucency of the composite, and refractive index ratio of the single components being used, in addition to the type and intensity of the curing light used and its distance from the material (22-26) .

CONCLUSION

The current study demonstrated that the addition of silver and zinc oxide nanoparticles to the sealants resulted in increasing the sealant hardness, and that the depth of cure (bottom to top ratio) qualified to the ADA specification number 39 for resin based fissure sealants for the two coloured sealants (grey and white).

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Table (3): Mean Microhardness Values For Fluoridated and Non Fluoridated Sealant

Type of Sealant	Microhardness Mean± SD
FS	7.64 ± 0.89
NFS	15.48 ± 0.56
T Value	-23.615
Significance	S

Table (4): Mean Microhardness Values For Both Sealants After the Addition

Type of Sealant	Microhardness Mean ± SD
FS	7.65 ± 0.89
ZnOFS	10.96±0.66
AgFS	14.49 ± 0.75
NFS	15.48 ± 0.56
ZnONFS	18.68 ± 0.96
AgNFS	22.22 ±1.01

Table (5): Microhardness Values of the Sealants Top Surface

Type of Sealant	Microhardness with out Addition of Nanoparticles Mean ± SD	Microhardness of Sealant with Zinc Oxide Mean ± SD	Microhardness of Sealant with Silver Mean ± SD
FS	7.65 c ± 0.89	10.96 b ± 0.66	14.49 a ± 0.75
NFS	15.48 c ± 0.56	18.68 b ± 0.96	22.22 a ± 1.03

Duncan's Multiple Range Test

Means with different letters are statistically significant horizontally
($p \leq 0.05$)

Table (6): Mean Microhardness for Both Sealants for Top and Bottom Surfaces

Sealant Type	Microhardness Top Mean ± SD	Microhardness Bottom Mean ± SD	T-Value	Significance
FS	7.64 ± 0.89	6.8 ± 0.92	2.074	**S****
ZnOFS	10.96 ± 0.66	9.36 ±0.82	4.83	**S***
AgFS	14.46 ± 0.75	11.78 ± 1.1	6.34	**S***
NFS	15.48 ± 0.56	14.35 ±0.98	3.161	**S***
ZnONFS	18.68 ± 0.96	16.88±1.01	4.013	**S***
AgNFS	22.22 ± 1.02	19.02± 0.93	7.254	**S***

S* Significant $p \leq 0.01$, **S**** Significant $p \leq 0.05$

Table (7): Hardness Ratio for All Groups of Sealants

Sealant Type	Mean Microhardness Top	Mean Microhardness Bottom	Bottom to Top Hardness Ratio
FS	7.64	6.8	0.89
ZnOFS	10.96	9.36	0.85
AgFS	14.46	11.78	0.81
NFS	15.48	14.35	0.92
ZnONFS	18.68	16.88	0.90
AgNFS	22.22	19.02	0.85