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Impact of Titanium Tetra Flouride Material and 810nm Diode Laser on Demineralized Human Enamel (In Vitro Study)

Amjed Kamel Al-Hassnawi[1], Nada Jafer MH. Radhi2*

(1) Ph. D. student, Department of Pediatric and Preventive Dentistry, College of Dentistry/University of Baghdad, Iraq.Specialist dentist in specialized dental center.Dewanyiah governorate.

(2) Department of Pediatric and Preventive Dentistry. College of Dentistry/University of Baghdad, Iraq.

***Corresponding author:** Nada Jafer MH. Radhi, **Phone:** (+964)7806314951, **Email:** nada.radhi@codental.uobaghdad.edu.iq

ABSTRACT

Background: Dental enamel was treated with laser irradiation and fluoride compounds as a method to enhance fluoride absorption by the enamel, which resulted in a rise in the mineral content of the enamel and a significant boost in its resistance to acid demineralization. **Objective:** The purpose of this research was to look into the effects of Titanium tetra Fluoride (TiF4) with or without diode laser on the demineralized enamel.

Material and methods: The sample consisted of 65 premolars teeth: 5 teeth remained sound for energy dispersive spectroscopy (EDS) analysis where 60 teeth were divided into 6 groups by treatment (N=10): (1) control negative (deionized water), (2) control positive (acidulated phosphate fluoride gel (APF)), (3) 6% Titanium tetra fluoride (TiF4) solution, (4) diode laser (810 nm, 0.5W, 60 s), (5) Laser then TiF4, and (6) Tif4 then Laser. Microhardness was measured before and after demineralization then after treatments. Five samples from each group were selected randomly for EDS analysis. **Results:** Microhardness increased significantly after all treatments ($p<0.05$). Results revealed a significant difference for all treatment groups ($p<0.05$). EDS analysis revealed that fluoride content was greater in 6%TIF4 then Tif4 with Laser groups, while the calcium content was greater in control positive followed by TiF4 groups.

Conclusion: TiF4 alone or in combination with laser enhancing demineralized enamel microhardness and fluoride content. Diode laser before or after TiF4 had minimal extra impact on remineralization.

Keywords: TiF4, Demineralized Enamel, Diode laser, Microhardness, Experimental study, University of Baghdad.

INTRODUCTION

Dental caries is one of the most common oral health issues, resulting in tooth demineralization caused by organic acids produced by biofilm bacteria from the metabolism of dietary fermentable carbohydrates, primarily sugars. The increase of resistance of teeth to acid attack can be achieved by fluoride that gives hardness and durability to the tooth enamel and protect against caries [(1)].

Titanium tetrafluoride (TiF4) has been studied since 1972 and has indicated promising preventive and therapeutic properties. It is reported as a stronger cariostatic agent compared to other fluoride compounds via formation of an acid-resistant coating composed of titanium dioxide or other organo-metallic complexes, and increased fluoride uptake due to the acidic nature of the compound [(2)].

Titanium tetra fluoride is metal fluorides, a nontoxic element, and no side effects have been reported with it. The benefit has been attributed to the compound's titanium group, which synergizes the impact of fluoride [(3)].

Laser irradiation has been used in the area of preventative dentistry for its potential to enhance tooth structure. Low-power red and near-infrared lasers seem to be a more appealing option for caries inhibition than high-power lasers. It has been shown that combining topical mineral treatments with laser irradiation has a synergistic impact on caries therapy and lowers enamel solubility and permeability [(4)].

Diode lasers, which are semiconductor lasers, produce coherent light of a single wavelength. Due to its compact size, low power consumption, and cheap cost of manufacture, diode lasers have become the most prevalent kinds of lasers in the world, being utilized in a wide range of components and disciplines of medical and dental operations [(5)].

When tissues interact with laser energy, the impact is affected by emission wavelengths, tissue optical characteristics, exposure period, laser intensity, and laser energy absorption into tissues. The absorptive effect governs how the atoms and molecules of the target tissue convert laser light energy into heat, chemical, acoustic, or non-laser light energy [(6)].

Laser irradiation is among the new techniques that might be promising in the field of remineralization [(7)] especially when applied with fluoride [(2)].

The impact of diode laser in conjunction with titanium tetrafluoride on demineralized enamel remineralization had not been explored before; hence the purpose of this research was to compare the potential effect of TiF4 alone or in combination with diode laser on demineralized enamel.

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MATERIALS AND METHODS

The sample:

An extracted upper first premolars teeth that collected from Iraqi private and governmental orthodontic centers .The teeth checked by 10X magnifying lens to being sound and free of any defect like hypoplasia, crack, carious lesions or filling. All the teeth were washed, polished using non fluoridated type of pumice slurry (8) and ultrasonicated to remove any pumice particals then placed in a plastic screw cup filled with de-ionized water, to which 0.1% crystals of thymol were added, in order to avoid bacterial growth. All the plastic cups was put in a fridge to avoid dehydration of teeth (9).

Enamel surface preparation:

The center of the buccal surface of each tooth was covered with a 5mm diameter circle of adhesive tape, while the other tooth surfaces were coated with acid resistant nail varnish to provide a paint free window on the buccal surface of each tooth once the adhesive tape was removed. Teeth were modified in an acrylic model (model size was 25 ×15 mm). For each paper size, grit papers (400,600, and 2000) were used to grind and polish each window 10 times in a circular manner. This method allowed for a flat surface on each tooth to be tested for microhardness (10).

Groups design:

The total sample of 65 teeth were divided randomly to 6 groups (A-F) each group consisted of ten teeth except the remaining 5 teeth stayed sound for chemical analysis by EDS. The groups subjected to 5 days of demineralizing and demineralizing solutions at 37°C to induce demineralized enamel surface (11). Each cycle consisted of a 3 hour immersion of each sample in 20ml of demineralizing solution at pH 4.3 (0.75 mM acetate buffer, 2.2 mM calcium chloride and 2.2 mM sodium phosphate) followed by a 21 hour immersion in remineralizing solution at pH 7.0 (20 Mm cacodylate buffer, 1.5 Mm calcium chloride, 0.9Mm sodium phosphate and 0.15 M potassium chloride). All the solutions were renewal every cycle (12).

After pH cycling and the sample demineralized, the 6 groups treated as followed:

Group (A): as a control negative had no treatment just in deionized water.

Group (B): as a control positive treated by acidulated phosphate fluoride gel 1.23% (ALPHA-PRO APF, USA) for four minutes by cotton applicator with dabbing movement on the windows of samples for 7 days (9).

Group (C): treated by immersing the samples separately in 6% TiF_4 solution 4 minutes, 1 time daily for 7 days.

Group (D): irradiated by diode laser (Quicklase,UK) in oscillatory semi contact motion around the surface for 1 minute for 7 days. The output power was 0.5 W with 810nm wavelength.

Group (E): irradiated for one minute as in group (D) then treated by immersing the samples separately in 6% TiF_4 solution 4 minutes, 1 time daily for 7 days.

Group (F): treated by immersing the samples separately in 6% TiF_4 solution 4 minutes then irradiated by diode laser for 1 minute as in group (D), 1 time daily for 7 days.

The TiF_4 solutions prepared by dissolving 6 gm solid TiF_4 powder in 100 ml deionized water according to concentration needed which was 6%. The pH of solution was about 4.5.

Energy dispersive spectroscopy (EDS) and micro hardness analysis achieved before and after demineralization by pH cycling then after treatment with materials. Energy dispersive spectroscopy (EDS) analysis device (Bruker, Germany) was used for examination the weight percentage (w%) of the following elements: O, P,Ca and F for 5 representative samples from each group selected randomly.

The micro hardness analysis achieved by digital micro-hardness tester (HVS-1000, Laryee, China) where each sample impressed with 300gm for 15 second in 3 places to do 3 indentations for each sample where the mean of these indentations were calculated for each sample (13).

Ethical approval:

This study was ethically approved on April 18, 2021 by the Institutional Review Board and Local Ethics Committee of the University of Baghdad (Document number 332). Written informed consent was obtained from all participants. This study was executed according to the code of ethics of the World Medical Association (Declaration of Helsinki) for studies on humans.

Statistical analysis

The collected data were introduced and statistically analyzed by utilizing the Statistical Package for Social Sciences (SPSS version -22, Chicago, Illionis, USA). Quantitative data were tested for normality by Kolmogorov-Smirnov test. Normal distribution of variables was described as means and SD, and One Way ANOVA was used for comparison between groups. P value ≤0.05 was considered to be statistically significant.

RESULT

Surface micro-hardness values of samples at base line, after demineralization and after treatments are listed in **Table 1**.

Table 1: Descriptive and statistical test of surface micro hardness among groups.

Groups	Baseline		Demineralization		Treatment		P value
	Mean	SD	Mean	SD	Mean	SD	
-Ve	271.120	20.768	47.592	6.254	28.240	13.816	0.000*
+Ve	281.780	29.266	46.380	4.056	66.880	32.245	0.000*
6% TiF4	281.280	47.059	52.064	5.958	113.560	14.332	0.000*
Laser	250.120	57.498	55.236	17.304	60.780	17.847	0.000*
L+ 6%TiF4	261.280	13.599	53.814	4.807	97.460	17.616	0.000*
6% TiF4 +L	268.640	13.515	61.770	14.576	95.460	24.714	0.000*
F	1.229		1.355		21.702		
P value	0.308		0.058		0.000*		

* Statistically significant difference at $p \leq 0.05$.

One way ANOVA indicated highly significant differences of all groups within the three phases: A base line, demineralization and treatments ($p<0.05$). Surface hardness of treatment phase had appeared significant differences of all groups while in base line and demineralized phases were not significantly different for all groups ($p>0.05$).The data obtained by (EDS) analysis showed that the mean weight for Ca ,F and P were reduced after demineralization while for O, the mean weight percentage was highly increased after demineralization .The maximum value for Ca was recorded with +Ve and 6%TIF4 groups as well as with Laser and L+6%TIF4 groups in less extent. The maximum value for F was recorded with 6%TIF4+L followed by 6%TIF4 then L+6%TIF4 groups. In relation to P, there was slight increase noticed after treatment with laser alone group as shown in **Table 2**.

Table 2: Descriptive and statistical test of weight percentage of elements among groups.

Elements	Groups	Mean	SD	P value
Oxygen	Sound	42.444	5.272	0.000*
	Demineralized	48.810	2.040	
	+Ve	17.444	4.034	
	6%TIF4	7.222	0.519	
	Laser	39.002	3.297	
	L+6%TIF4	11.404	2.363	
	6%TIF4+L	14.552	1.646	
P	Sound	17.252	3.784	0.000*
	Demineralized	10.004	1.825	
	+Ve	6.096	1.790	
	6%TIF4	0.976	0.391	
	Laser	10.784	1.552	
	L+6%TIF4	2.086	1.052	
	6%TIF4+L	4.246	0.751	
Ca	Sound	33.594	8.050	0.000*
	Demineralized	24.012	4.521	
	+Ve	42.530	6.749	
	6%TIF4	35.450	3.117	
	Laser	30.122	5.106	
	L+6%TIF4	31.626	2.843	
	6%TIF4+L	18.412	1.024	
F	Sound	.050	.039	0.000*
	Demineralized	0.020	0.028	
	+Ve	8.278	2.105	
	6%TIF4	45.352	3.635	
	Laser	0.150	0.127	
	L+6%TIF4	38.740	3.424	
	6%TIF4+L	48.128	1.789	

*Statistically significant difference at $p \leq 0.05$.

DISCUSSION

In this study, Acidulated Phosphate Fluoride (APF) was selected as a positive control for evaluation of new and especially acidic fluoride compounds (Titanium tetra Fluoride (TiF4) in present study) since it is known to be a potential remineralizing agent of initial caries. The acidic nature of this compound causes increased fluoride uptake and more production of fluorohydroxyapatite during its chemical reaction with tooth mineral [(14)].

The microhardness findings demonstrated the impact of 6% TiF4 alone or in conjunction with an 810nm diode laser on the microhardness and chemical structure of demineralized human enamel of permanent teeth. The microhardness examination was done in three stages: the first as a baseline, then tooth immersion in the demineralizing solution, and finally following the application of various treatment procedures. The absence of a significant difference in base line and demineralized stages across groups supported the existence of similar enamel demineralization in the research groups. However, all groups showed a substantial change in microhardness following treatment. The use of TiF4 alone, followed by laser irradiation, was the most successful technique for increasing the microhardness of demineralized surfaces, followed by control positive (APF). The energy dispersive spectroscopy (EDS) analysis of the study groups confirmed the results of surface micro-hardness test where the maximum value for F was recorded with 6%TIF4+L followed by 6%TIF4 then L+6%TIF4 groups. The maximum value for Ca was recorded with 6%TIF4 groups as well as with Laser and L+6%TIF4 groups in less extent. Since the Ca and F content were responsible about remineralization and increased the hardness of the enamel, the sum of two element in 6%TIF4 group was the maximum followed by L+6%TIF4, 6%TIF4+L then +Ve and lastly laser alone groups.

The titanium and fluoride ions of TiF4 were responsible for its efficacy by forming protective mechanical and chemical barriers against noxious agents that harm the tooth surface [(15)]. In the aqueous environment, TiF4 decomposes and changes into titanium dioxide (TiO2) which made a resistant glaze-like layer on the tooth surface (mechanical protection), and the acidic compound of HF that resulted in some surface porosities that increase the fluoride uptake (chemical protection) on the tooth surface [(16)]. Like other fluoridated agents, the fluoride ion in TiF4 adheres to the calcium ions of the tooth surface and form CaF2 globules which replace hydroxyl ions released from the tooth structure during acidic challenges. The resultant fluoroapatite crystals are more acid resistant than hydroxyapatite [(17)]. The better fluoride uptake after application of TiF4 is related to the ability of its polyvalent metal ions to form organometallic complexes of fluoride that are strongly bonded to enamel hydroxyapatite molecules [(18)].

According to results of the present study, the effectiveness of TiF4 alone or combined with laser (before or after) was differed to the positive control group (APF), although they were all more effective in remineralization of the demineralized enamel compared to the control negative group. These results are agreed to the results of **Exterkate *et al***. who reported more effectiveness of TiF4 compared to NaF in caries prevention since they used NaF as control positive [(18)]. The results of present study disagreed to the results of **Comar *et al.*** and **Alcantara *et al.*** that indicated the similar effectiveness of TiF4 and sodium fluoride (NaF) in remineralization of the initial caries lesions [(19,20)]. The difference in results may be related to the differences in the positive control materials, due to depth of lesions or frequency and concentration of TiF4.

The combination therapy between laser and fluoride was reported more beneficial for caries prevention than laser irradiation or fluoride therapy alone. Laser irradiation of fluoridated tooth causes formation of a-tricalcium phosphate (TCP), fluoroapatite [(21)] and Tetracalcium diphosphate that increasing the strength of tooth enamel [(22)].

It is proposed that if a laser is used after or through a fluoride compound, the heat produced by the laser aids in the fusing of the loosely-attached intermediate CaF2 crystals created on the tooth surface [(23)] and also transforms hydroxyapatite into fluoroapatite in the presence of fluoride ion [(24)].

Previously, lasers with high power like CO2, Er:YAG or Nd:YAG lasers were employed to improve enamel resistance to caries assault by melting, fusing, and resolidification of enamel crystals. Another process responsible for greater acid resistance is the decrease of enamel's water and carbonate content, which leads to an increase in hydroxyl ion concentration and the breakdown of enamel proteins [(4)].

The diode laser 810 nm has a limited absorption band in dental enamel, however as compared to healthy enamel; the demineralized and weak surface enhanced the action of the laser. The laser power employed in this investigation was 0.5W, which is on the line between high and low power of lasers. When compared to positive controls, the use of TiF4 in conjunction with laser irradiation was the most successful in increasing the microhardness of demineralized surfaces (APF). This might be owing to the semi-contact application and the accumulative impact of laser for seven days with a synergistic effect with TiF4.

The findings of this investigation contradicted those of **Santaella *et al.*** [25], who discovered that fluoride varnish was more successful than diode laser treatment in improving sound enamel resistance. This research also contradicted the findings of **Ahrari *et al.*** [4], **Bahrololoomi *et al.*** [26], and **Kato *et al.*** [27].

The current study's findings were partially consistent with those of **Fekrazad *et al*.**, who discovered that TiF4 before CO2 laser irradiation significantly increased the microhardness of initially demineralized enamel surfaces, but that CO2 laser irradiation before TiF4 application could not remineralize the white-spot lesions [7].

Changes in laser parameters such as power, wavelengths, pulse frequencies and time of irradiation may produce varied outcomes, hence more research is recommended.

CONCLUSION

The present study was concluded that TiF4 alone or in combination with diode laser increased the micro-hardness of demineralized surfaces of enamel significantly in comparison to APF. These methods can be recommended as useful treatment protocols for demineralized lesions.

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Conflict of Interest: Nil.

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