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Evaluation of Bioactive Nano Composite Fillers Effect on Wear Resistance of Composite and Enamel Surfaces

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A B S T R A C T

The aim of this study was to evaluate the wear resistance of recent commercially available dental composites. Tested composite samples were divided into two groups. Each group involved 20 prepared composite discs. For antagonist, enamel samples, 20 premolar buccal cusps were selected, prepared and embedded in acrylic mold. The two body wear testing was performed using a programmable logic controlled equipment. Surface roughness evaluated using digital microscope and images analysis. Data analysis was performed using Student t-test and Aasistat 7.6 statistics software in experimental composite groups, It was found that group B composite recorded statistically significant higher ($p=0.009 < 0.05$; Weight loss mean value than group A composite mean value). Also group B composite recorded higher, statistically non-significant ($p=0.09 >$), roughness change mean value than group A composite mean value. In antagonistic cusp groups, it was found that group B antagonistic cusp recorded, statistically non-significant, higher weight loss mean value than group A antagonistic cusp mean value. Also group A antagonistic cusp recorded, statistically non-significant, higher roughness change mean value than group B antagonistic cusp mean. Nanoparticles zirconia resin composite showed high wear resistance and bioactive composite showed a clinically accepted wear resistance.

Introduction

Recently dental resin composites are considered as perfect option for treatment all types of restorations Kurachi *et al.*, (2001), Pontons-Melo *et al.*, (2012) and Bartlett *et al.*, (2006).

Application of direct and indirect resin composites takes place to build up the occlusion in cases of extensive tooth wear Attin *et al.*, (2012), Pontons-Melo *et al.*, (2011), Vailati *et al.*, (2012) and Ferracane (2013).

The reason of teeth wear (bruxism, erosion or combination of both), will effect on success or failure of resin composite dental restorations. As failure of direct resin composite represented by wear, fracture and recurrent caries Vailati *et al.*, (2012) and Ferracane (2013), Wilder (1999) and Da Rosa Rodolpho *et al.*, (2006).

Nowadays, polymer matrix was dispersed by nanoparticles in hybrid system of dental resin

composites, this system was received extensive attention from dentists (Lai *et al.*, 2007; Mitr, 2003).

Wear resistance is one of the most important of mechanical properties of restorative materials, which determines the success or failure and shelf life of resin composites (Suwannaroop *et al.*, 2011).

Normally in oral cavity, Restorative material wear results from direct contact between the tooth and the restorations during mastication, parafunctional stresses, and abrasive particles tooth brushing and dietary factors which introduce chemical factors to oral media (Braga *et al.*, 2010; Hahnel *et al.*, 2009).

Wear of dental restorations will be a main reason in structure alteration which may result in loss of vertical dimension of occlusion with subsequent teeth occlusion alteration and faulty tooth relationship with esthetic loss.

Lack of sufficient wear resistance will result in excessive reduction in structure, resulting in loss of posterior tooth support, loss of masticatory efficiency, alterations in the functional path of masticatory movement, fatigue of masticatory muscles (Suwannaroop *et al.*, 2011; Ghazal *et al.*, 2008; Hirata, 2011).

Nanoparticles used in biomedical dental restorative materials to improve mechanical properties and increase wear resistance of used material (Xia *et al.*, 2008; Tian *et al.*, 2008).

The load transfer from polymer matrix to nanoparticles is enhanced through large specific nanoparticles (size<100 nm) surface area than microparticales (size>100 nm) (Mitra, 2003). Therefore, in this study we hypothesize that these nanoparticles might improve wear resistance of tested nano bioactive composites. The objective of this study was to investigate the effect of nano bioactive particles on wear resistance of tested materials against natural teeth.

Materials and Methods

Two direct commercially available dental nanocomposites were tested in this study, their manufacturers, composition and lot number are summarized in table 1.

Composite samples were divided into two group as follow:

Group A representing 20 discs of Filtek Z350 XT and
Group B representing 20 discs of Bio active restorative.

Samples preparation

Twenty disc shaped (10 mm diameter x 2mm thick) samples of each tested nanocompsite materials group were prepared according to the manufacturer's instructions.

Each type of tested resin composite was inserted in a cylindrical Teflon mold (10 mm diameter x 2mm thick) and backed with polyester strip (Mylar, Moyco Union Broach, York, PA, USA). The top surface of the mold was then covered with another polyester strip.

Filtek Z350 XT and Bio active restorative were cured for 40 s using a conventional halogen light curing unit, which had a light intensity of 450–470 mW/ cm2 (Monitex, Blue LEX, LD-105, Taiwan) (Fig. 1).

For Antagonist Samples, Twenty upper Premolar halves (Sound non carious buccal Cusps) were selected and prepared. Premolars buccal cusps were embedded in circular acrylic block with 20 mm diameter 25, mm height and fixed to wear test machine used as illustrated in figure 2.

Two-body wear test

The two body wear testing was performed using a programmable logic controlled equipment; the newly developed four stations multimodal ROBOTA chewing simulator integrated with thermo-cyclic protocol operated on servo-motor (Model ACH-09075DC-T, AD-Tech Technology Co., Ltd., Germany).

ROBOTA chewing simulator which has four chambers simulating the vertical and horizontal movements simultaneously in the thermodynamic condition. Each of the chambers consists of an upper Jackob's chuckas tooth antagonist holder that can be tightened with a screw and a lower plastic sample holder in which the specimen can be embedded. The composite specimens were embedded in Teflon housing in the lower sample holder (Fig. 3). A weight of 5 kg, which is comparable to 49 N of chewing force was exerted. The test was repeated 10, 000 times to clinically simulate the 1 month chewing condition, accompanying thermocycling according to previous studies (Table 2) (Yu-Seok, 2010).

The substance loss of the specimens after loading was measured by weighting in the electronic analytical balance (Sartorius, Biopharmaceutical and Laboratories, Germany) with an accuracy of 0.0001 gr. to weight the difference in weight before and after 37, 500 cycles. As this electronic balance had a fully automated calibration technology and a micro weighting scale, values of all the mounted discs and antagonist samples were accurately measured. Each mounted sample was cleaned and dried with tissue paper before weighing. To ensure accuracy, the balance was kept on a free standing table at all times - away from vibrations - and weighed the specimens with the glass doors of the balance closed to avoid the effect of air drafts (Fig. 4).

Roughness methodology

The optical methods tend to fulfill the need for quantitative characterization of surface topography without contact (Ossama *et al.*, 2010). Specimens were photographed using USB digital microscope with a built-in camera (Scope Capture Digital Microscope, Guangdong, China; Fig. 4) connected with an IBM compatible personal computer using a fixed magnification of 120X. The images were recorded with a resolution of 1280 × 1024 pixels per image. Digital microscope images were cropped to 350 x 400 pixels using Microsoft office picture manager to specify/standardize area of roughness measurement. The cropped images were analyzed using WSxM software (Ver5 develop 4.1, Nanotec, Electronica, SL) (Horcas *et al.*, 2007). Within the WSxM software, all limits, sizes, frames and measured parameters are expressed in pixels. Therefore, system calibration was done to convert the pixels into absolute real world units. Calibration was made by comparing an object of known size (a ruler in this study) with a scale generated by the software. WSxM software was used to calculate average of heights (Ra) expressed in μm, which can be assumed as a reliable indices of surface roughness (Kakaboura, 2007).

Subsequently, a 3D image of the surface profile of the specimens was created using A digital image analysis system (Image J 1.43U, National Institute of Health, USA) (Fig. 5).

Statistical analysis

Data analysis was performed in several steps. Initially, descriptive statistics for each group results. Student t-test was performed to detect significant difference between groups. Statistical analysis was performed using Aasistat 7.6 statistics software for Windows (Campina Grande, Paraiba state, Brazil). P values ≤ 0.05 are considered to be statistically significant in all tests.

Results and Discussion

Wear

The mean values and standard deviations (SD) for wear measured by weight loss (gram) recorded on both materials before and after wear simulation cycles summarized in table 3 and graphically represented in figure 5. The wear recorded for the antagonistic cusps is also shown.

Weight

In experimental composite groups

It was found that group B composite recorded higher weight loss mean value (0.00592±0.0018 gr) than group A composite mean value (0.00027±0.0004gr).

The difference between both groups was statistically significant as indicated by t-test ($p=0.009 < 0.05$) (Table 4).

In antagonistic cusp groups

It was found that group B antagonistic cusp recorded higher weight loss mean value (0.01±0.002 gr) than group A antagonistic cusp mean value (0.0049±0.005gr).

The difference between both groups was statistically non-significant as indicated by t-test ($p=0.2947 > 0.05$) (Table 4).

Roughness

The mean values and standard deviations (SD) for roughness measured by average roughness Ra (µm) recorded on both materials before and after wear simulation cycles and summarized in tables 5 and 6. It is graphically represented in figures 6–12. The roughness recorded for the antagonistic cusps is also shown.

In experimental composite groups

It was found that group B composite recorded higher roughness change mean value (0.00079±0.0007 Ra) than group A composite mean value (-0.00067±0.0003 Ra). The difference between both groups was statistically non-significant as indicated by t-test ($p=0.09 > 0.05$).

In antagonistic cusp groups

It was found that group A antagonistic cusp recorded higher roughness change mean value (0.00077±0.0006 Ra) than group B antagonistic cusp mean value (-0.00057±0.0004 Ra). The difference between both groups was statistically non-significant as indicated by t-test ($p=0.0537> 0.05$).

Development of bioactive dental composite restorations requires clinical and laboratory evaluation techniques to permit assessment of its mechanical clinical properties coincide with its biological properties. Bioactive dental composite surface wear is an important mechanical clinical property to be investigated.

DeLong *et al.,* (2012) mentioned that dental composites wear measuring assume that occlusal forces and contact paths, which are highly variable both within and between subjects, can be represented by average values that remain relatively stable with time.

Recent dental restoration and Natural teeth wear resistance are an important property to be studied. Absence of wear resistance can be a major cause of vertical dimension loss with subsequent of temporo-madibular joint dysfunction. This was clear in patients with para-functional pathology *e.g.* bruxism and clenching. That can lead to myofacial muscle dysfunction, pain and headaches. Also reaching healthy oral cavity equilibrium will be difficult (Olivera, 2008).

Table.1 Composition, lot number and manufacture of the tested materials

Material	composition	Lot number	Manufacture
Filtek Z350 XT	(20 nm silica filler 4-11 nm zirconia filler) as 72.5% by w filler bis-GMA, UDMA, TEGDMA, PEGDMA and bis-EMA resins	N339145	3M ESPE
Bio active restorative	56% by weight reactive glass particles that mimic physical and chemical properties of natural teeth., shock absorbing ionic resin component containing acidic monomer with antimicrobial properties.no Bisphenol A, No BisGMA, no BPA derivatives'	150318	PULPDENT Corporation

Table.2 Wear test parameters

Cold/hot bath temperature: 5°/55℃	Dwell time: 60 s
Vertical movement: 1 mm	Horizontal movement: 2 mm
Rising speed: 90 mm/s	Forward speed: 90 mm/s
Descending speed: 40 mm/s	Backward speed: 40 mm/s
Cycle frequency 1.6 Hz	Weight per sample: from 5 kg
Torque; 2.4 N.m	

Table.3 Weight results (Mean values ±SD) for both experimental groups and cusp antagonist before and after wear simulation

Variables		Samples		Antagonistic cusp	
		Before	After	Before	After
Composite group	Group A	0.1577±0.003	0.1572±0.004	0.5524±0.062	0.5475±0.056
	Group B	0.1590±0.001	0.15308±0.006	0.5119±0.0527	0.5019±0.0516

Table.4 Weight loss results (Mean values ±SD) for both experimental groups and antagonist as function of wear simulation

Variables		Samples			Antagonistic cusp		
		Mean±SEM	95 % CI		Mean±SEM	95 % CI	
			Lower	Upper		Lower	Upper
Composite group	Group A	0.00053±0.0002	0.0001	0.001	0.0049±0.005	-0.0064	0.01624
	Group B	0.00592±0.0018	0.0018	0.01	0.01±0.002	0.0048	0.0153
ANOVA	P value	0.009*			0.2947 ns		

CI; Confidence intervals*; significant ($p<0.05$) ns; non-significant ($p>0.05$)

Table.5 Roughness results (Mean values ±SD) for experimental groups and cusp antagonist before and after wear simulation

Variables		Samples		Antagonistic cusp	
		Before	After	Before	After
Composite group	Group A	0.2557±0.0009	0.2550±0.0007	0.2555±0.0013	0.2563±0.001
	Group B	0.2555±0.0008	0.2562±0.0015	0.2567±0.0008	0.2561±0.0007

Table.6 Roughness change results (Mean values ±SD) for both experimental groups and antagonist as function of wear simulation

Variables		Samples			Antagonistic cusp		
		Mean±SEM	95 % CI		Mean±SEM	95 % CI	
			Lower	Upper		Lower	Upper
Composite group	Group A	-0.00067±0.0003	-0.0016	0.0002	0.00077±0.0006	-0.0006	0.0022
	Group B	0.00079±0.0007	-0.0008	0.0024	-0.00057±0.0004	-0.0016	0.0005
ANOVA	P value	0.091 ns			0.0537ns		

CI; Confidence intervals*; significant ($p<0.05$) ns; non-significant ($p>0.05$)

Fig.1 prepared tested nanocompsite materials

Fig.2 Antagonist enamel samples illustration

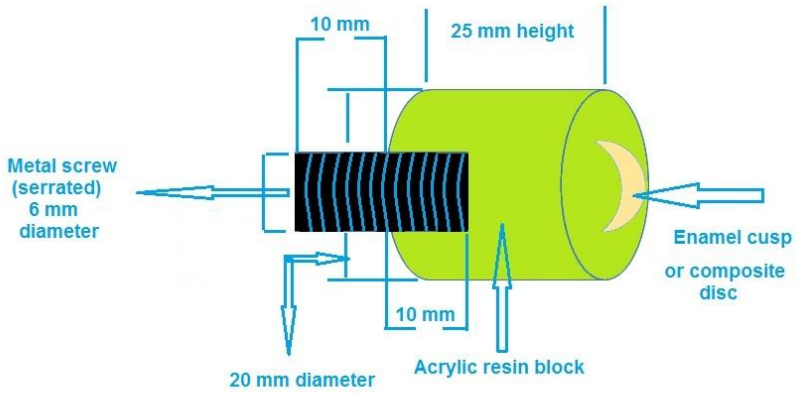


Fig.3 ROBOTA chewing simulator

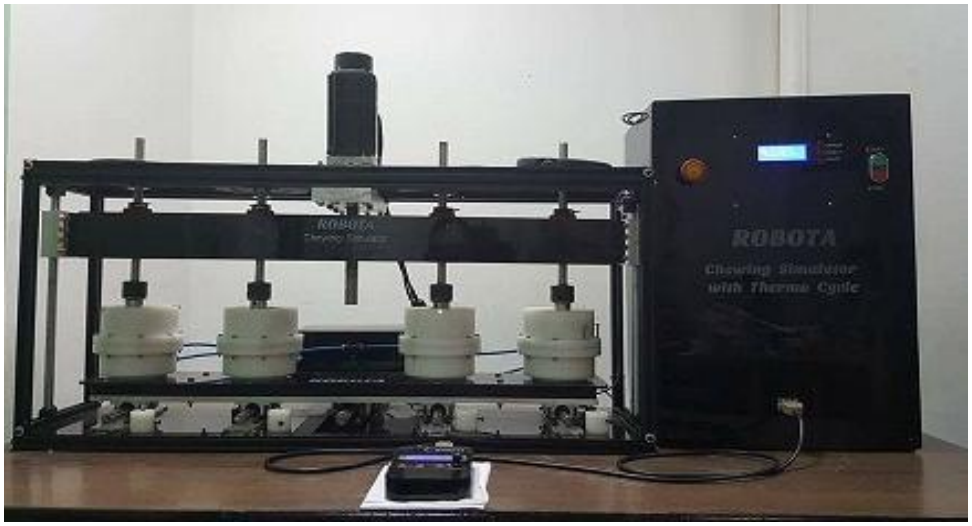


Fig.4 Electronic analytical balance

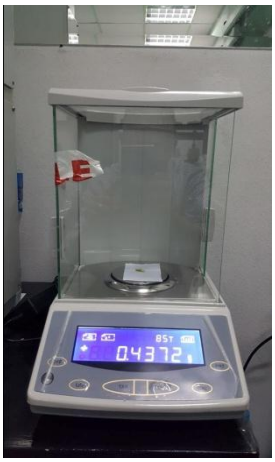
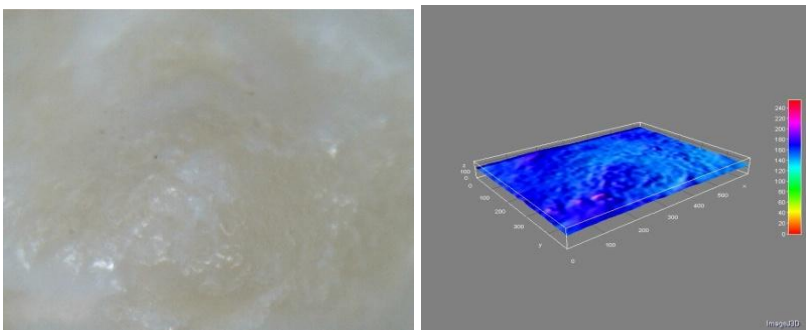


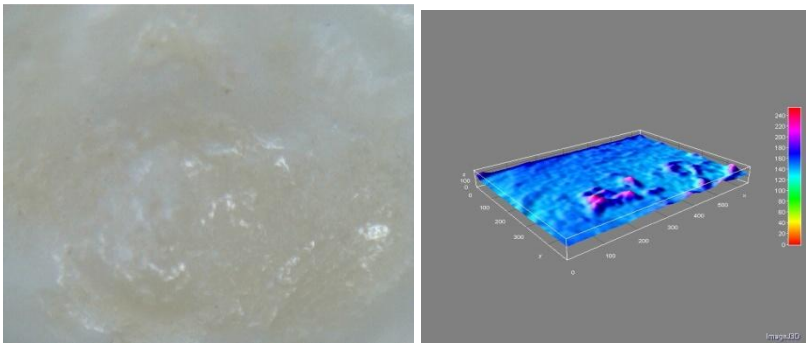
Fig.5 Scope capture digital microscope

Fig.6 Group A and B reprehensive sample of antagonist cusp surface roughness before wear process. I- Antagonist buccal cusp micrograph II- sample surface plot



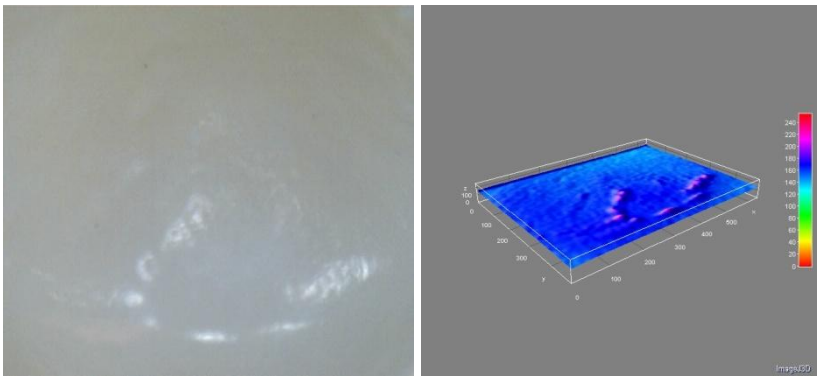
I II

Fig.7 Group A antagonist cusp surface roughness after wear process. I- Antagonist buccal cusp micrograph II- sample surface plot



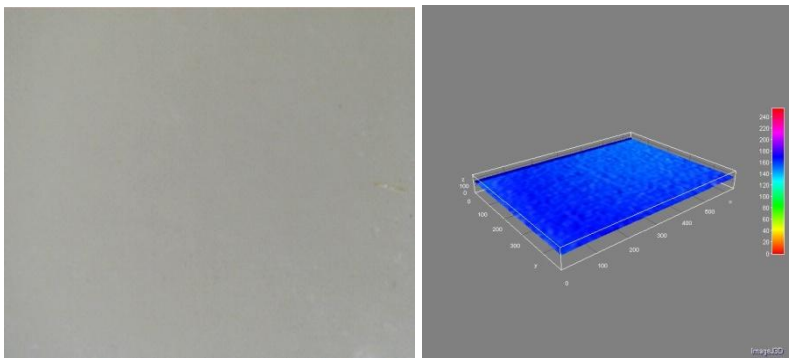
I II

Fig.8 Group B antagonist cusp surface roughness after wear process. I- Antagonist buccal cusp micrograph II- sample surface plot



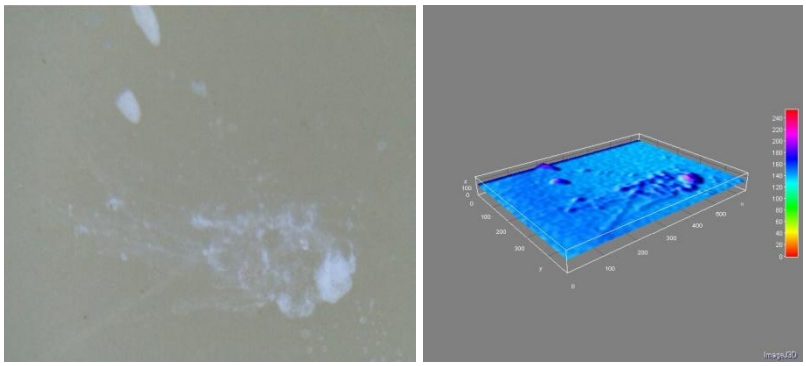
I II

Fig.9 Group A composite samples surface roughness before wear process.
I- Composite surface micrograph II- Composite sample surface plot



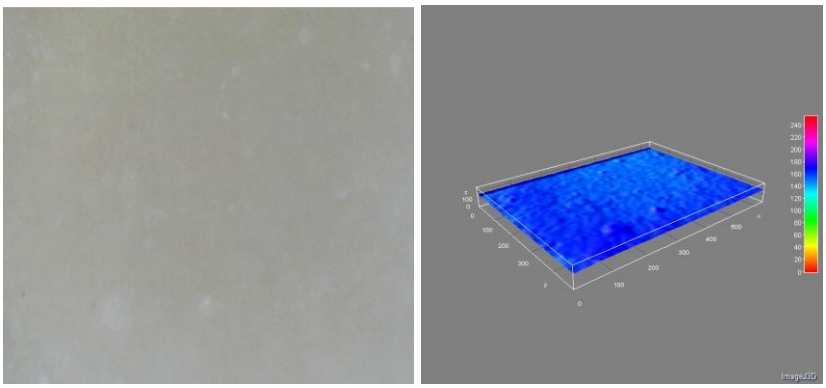
I II

Fig.10 Group A composite samples surface roughness after wear process.
I- Composite surface micrograph II- Composite sample surface plot



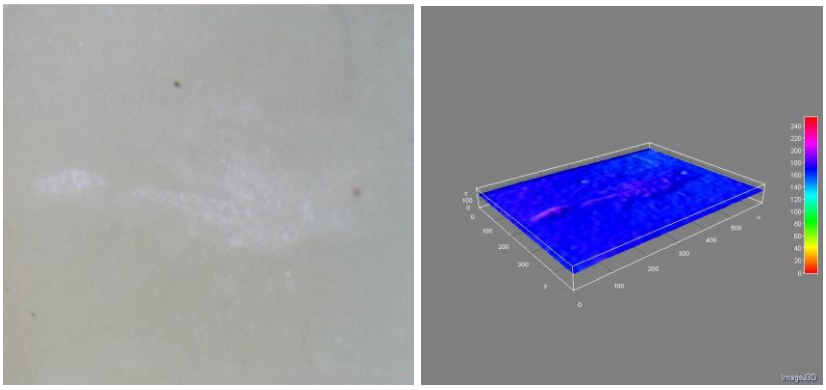
I II

Fig.11 Group B composite samples surface roughness before wear process.
I- Composite surface micrograph II- Composite sample surface plot



I II

Fig.12 Group B composite samples surface roughness after wear process. I- Composite surface micrograph II- Composite sample surface plot



I II

In present study, a two body wear test was conducted to rank the wear resistance of different recent resin composite materials. The pairs of human tooth- Filtek Z350 XT composite samples and human tooth - Bio active restorative materials samples have been subjected to a wear test protocol in this study.

In this study it was found group B composite samples showed non-significant higher surface roughness than group A. and its antagonist cusp showed non-significant higher weight loss than group A antagonist cusp. It was found that group B composite recorded higher was statistically non-significant roughness change mean value (0.00079±0.0007 Ra) than group A composite mean value (-0.00067±0.0003 Ra). Also it was found that groupA antagonistic cusp showed non-significant higher roughness change mean value than group B antagonistic cusp mean value.

Our findings may be explained as higher enamel weight loss and subsequent wear of tooth antagonist to group B with surface roughness of group B samples caused by glass particles and wear debris that detach during the wear process might behave as an abrasive medium and lead to a 3-body wear mechanism. These findings were coinciding with previous studies that confirmed that these abrasive particles might emphasize the consequences of enamel wear. Although this wear test was run using distilled water, which would help lubricate the contact surface, flush out debris, and reduce heat generation from abrasion, some wear debris may still remain in the wear track and may influence the contact stresses and wear (Fischer *et al.,* 2000; Shimane *et al.,* 2010; Sripetchdanond *et al.,* 2014).

Group A antagonist cusp showed non-significant higher roughness change mean value, this may attributed to a harder filler, with high filler load, becomes less abrasive when the particle size is at nano-scale. Nanoparticle Zirconia filler used in group A tested composite inherited it to be less abrasive than glass particles filler, with lower filler load, tested with group B.

These findings matched with several researchers who stated that harder filler becomes less abrasive when the particle size is at nano-scale. The use of hard filler with a large size should be avoided. Though the configuration of the fillers becomes evident on the SEM pictures made after the two and three-body wear tests differences in roughness cannot be found with the profilometer (Ilie *et al.,* 2009; Ruttermann *et*

al., 2008).

It was found that group B composite recorded statistically significant higher weight loss mean value (0.00592±0.0018 gr) than group A composite mean value (0.00027±0.0004gr). Also It was found that group B antagonistic cusp recorded non-significant higher weight loss mean value (0.01±0.002 gr) than group A antagonistic cusp mean value (0.0049±0.005gr). This weight loss of group B composite samples and antagonist cusp may be due to glass particles leaching out of tested composite samples and these particles act as abrasive medium. The abrasive effect of leached glass partials extends to abrade opposing dentition with subsequent weight loss. As in dental composites wear mechanism, the fillers type plays an important role. Faria *et al.,* (2007) mentioned that during clinical application of composites silica-filled composite has been recommended instead of a glass-filled composite that leaches more filler elements and degrades faster. For tested composite samples group A which show statistically significant lower weight loss than group B, Also group A antagonist showed non statistically significant lower weight loss values than Group B antagonist. These may be explained by the composition of group A tested composite which contain 72.5 % by weight nanoparticles Zirconia filler immersed in rein matrix formed mainly of bis-GMA, UDMA, TEGDMA, PEGDMA and bis-EMA resins. It was found that smaller particles with high filler friction volumes showing high wear resistance. The inter-particle spacing decreasing was the key to improving the wear resistance of composites. Dental Composites inter-particle spacing decreasing could be reduced by both decreasing the size and increasing the volume fraction of the fillers (Lim *et al.,* 2009; Manhart *et al.,* 2000).

Within the limitations of this study, the following conclusions can be drawn:

The use of tested bioactive composite was showed a clinically accepted wear resistance with minimal alteration of opposing natural teeth tissue.

Nanoparticles zirconia resin composite tested show high wear resistance with minimal surface topographical changes of natural teeth antagonist.

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