Anoj Meena, Harlal Singh Mali, Amar Patnaik and Shiv Ranjan Kumar*

Investigation of wear behavior of nanoalumina and marble dust-reinforced dental composites

<https://doi.org/10.1515/secm-2017-0152>

Received April 30, 2017; accepted May 24, 2018; previously published online August 14, 2018

Abstract: In the present work, the effects of adding nanoalumina and marble dust on the wear behavior of dental composites were investigated. The hardness of dental composite was determined using Vickers micro-hardness tester. A two-body abrasive wear test was performed on the dental wear simulator under the medium of artificial saliva. The experiments were performed as per the Taguchi orthogonal array and steady state condition by varying parameters such as filler content, normal load, sliding velocity, and number of cycles. The hardness results indicated that the incorporation of 5 wt. % of nanoalumina increased the hardness of the dental composite by 12%, whereas the incorporation of 5 wt. % of marble dust increased the hardness of the dental composite by 7%. Also, for the experiments as per the Taguchi orthogonal array, the mean volumetric wear in the case of nanoalumina-filled dental composite was 9.6% less than that of marble dust-filled dental composite. However, in both the cases, the volumetric wear increased with the increase in normal load, sliding speed, and number of cycles but decreased with the increase in filler content. Analysis of variance (ANOVA) of the results indicated that normal load was less significant compared to filler content, sliding speed, and number of cycles.

Keywords: dental composite; marble dust; nanoalumina; Taguchi method; wear.

1 Introduction

The study of wear behavior of dental materials is one of the important factors in understanding the deterioration level under clinical conditions. The excessive differential wear of teeth and restorative materials has significant deleterious effects on the biologic, functional, and aesthetic condition of the masticatory system. Hence, in this

regard, the controlled investigations of various factors that might influence tooth and restoration wear have been performed to date. These examinations include the study of the effects of thermo-cycling [1], cyclic loading [2, 3], applied load [4], degree of polymerization [5], aging [6], water absorption [7], lubricant pH [8, 9], surface coatings [10, 11], tooth brushing/dentifrices [12], and silanization [13–15] on the wear of dental material. The wear assessments for these studies were based on two-body and three-body wear simulation methods. However, the major loss in dental material is due to abrasion and erosion phenomena in the oral environment. It has been documented that the effect of filler volume on wear resistance follows a linear relationship, with high filler volumes decreasing wear rates [16]. In dental implantology, the alumina was initially used by the dental material scientist mainly in screws, anchorage elements, pin implants, and crowns, etc. [17, 18]. The application of alumina as a filler in dental materials provided appropriate aesthetic properties, chemical and biological inertness, high mechanical wear resistance, and overall long-term stability [19].

Currently, most commercial dental composite resins contain inorganic nanoparticles to achieve enhanced performance. These improvements are suggested to arise from the high specific surface area and rich surface functional groups of the nanoparticles [20]. Although these nanoparticles have substantially improved composite properties including abrasion resistance, polymerization shrinkage, and mechanical strength, more progress is still needed to achieve long-term satisfactory restorations for clinical therapy [21, 22]. In Rajasthan, India, one of the serious concerns is how to utilize marble dust or slurry. A very large amount of marble is being dumped by marble cutting industries in any nearby pit or vacant spaces. This imposes the biggest threats to nature and human health. Simultaneously, it produces challenges to the environment in terms of the topographic alteration, land occupation, surface and ground water contamination, and air pollution [23–25]. Therefore, utilization of marble dust in the production of new materials will help to protect the environment. Recently, the use of marble dust as replacement materials was investigated by various researchers [26–30]. Therefore, recycling and reuse of industrial wastes play a vital role in both solving the industrial waste problem and in getting benefit from it. Marble dust is used as one

*Corresponding author: Shiv Ranjan Kumar, Mechanical Engineering Department, JECRC University, Jaipur, India, e-mail: ranjan.shiv@gmail.com

Anoj Meena, Harlal Singh Mali and Amar Patnaik: Mechanical Engineering Department, M.N.I.T Jaipur, India

of filler ingredients that can adequately and reliably contribute to the performance of dental composite materials. Apart from the performance aspect, marble dust will greatly reduce the cost of artificial teeth materials.

The aim of the current study was to evaluate the wear performance of dental composite materials filled with nanoalumina and marble dust filler in an artificial saliva medium under steady state conditions and the Taguchi experimental design.

2 Materials and methods

2.1 Materials

Two different particulate filler materials such as nanoalumina and commercially available marble dust were used in this study. The properties of nanoalumina (particle size 20 nm, high purity 98%) and marble dust (particle size 70 mm, Grade Makrana Marble, Udaipur, India) are presented in Tables 1 and 2, respectively. Monomer bisphenol-A glycidyl methacrylate (BisGMA) was procured from Esstech Inc. (Essington, PA, USA). The diluent tri-ethylene glycol dimethacrylate (TEGDMA) was purchased from TCI (Tokyo, Japan). Photo initiator camphorquinone (CQ) was purchased from Spectrochem (Mumbai, India). 2-dimethylaminoethyl methacrylate (DMAEMA), butylated hydroxytoluene (BHT) and coupling agent methacryloxy propyltrimethoxy silane (MPS) were purchased from Sigma Aldrich (Bangalore, India). For two-body wear assessment, artificial saliva was used, and the composition is presented in Table 3.

2.2 Fabrication of dental composite

The matrix material consists of resin matrix, e.g. BisGMA (52 wt. %) and TEGDMA (45 wt. %) and were mixed

Table 3: Medium used for wear analysis in the present study.

Medium	Marketed by	Brand name	Composition
Artificial saliva	Global Dent Aids Pvt. Ltd., New Delhi, India	Saleva	Water, sorbitol, glycerin, gum, sodium saccharin, sodium phosphate, cetylpyridinium chloride, brilliant blue

together with the polymerization initiator and accelerator CQ, DMAEMA, and BHT. Initially, the fillers nanoalumina and marble dust were mixed with silane (MPS) and water solutions and stirred uniformly up to 6 h. Nine different composites were prepared by varying weight percentages of silane-treated nanoalumina and marble dust, respectively. Composites were prepared by pouring a blend mixture into a mold of the desired dimensions. The compositions of composite resins are presented in Table 4.

2.3 Vickers micro-hardness test

The nanoalumina- and marble dust-filled dental composite specimens of dimensions 8-mm diameter and 6-mm length were fabricated in the glass mold. After curing was done, the specimens were placed in distilled water for 24 h to ensure complete curing. Then, the specimens were subjected to 500-g load up to 30-s test on the machine micro-hardness tester (UHL VMHT, Walter, UK). Six indentations were made on the surface of the sample. The mean value (d) of the six indentations on each sample was considered. The Hv values were calculated in accordance to the ASTM E384-11e1 standard using Eq. (1).

$$Hv = 1854.4 \times \frac{F}{d^2} \quad (1)$$

Table 1: Properties of aluminum oxide nanoparticles.

Properties	Metric
Density	3.85 g/cm ³
Molar mass	101.96 g/mol
Melting point	2100°C

Table 2: Properties of marble dust.

Properties	Metric
Fineness/fineness modulus	1.5 m ² /g
Specific gravity	2.67

Table 4: Detailed composition of unfilled and particulate-filled dental composites.

Material	Resin	Filler content	Filler type and size
DC-0	BisGMA (52 wt. %),	0 wt. %, Alumina: 40–70 nm	
DCAL-5	TEGDMA (45 wt. %),	5 wt. % spherical, density	
DCAL-10	(2% BHT), CQ (0.2 wt. %)	10 wt. %, 3.95 g/ml	
DCAL-15	and DMAEMA (0.8 wt. %)	15 wt. %, 20 wt. %	
DCAL-20		20 wt. %	
DCMD-5		5 wt. %, Marble dust	
DCMD-10		10 wt. %, powder: 20–30	
DCMD-15		15 wt. %, μm, density 2.6	
DCMD-20		20 wt. % g/cm ³	

2.4 Wear characterization

Ducom dental wear simulator (Ducom, Bangalore, India) was used to evaluate the tribological characterization in the presence of an artificial saliva environment. Dental wear simulator is compatible to conduct tests according to the ASTM F732 test procedure. It was used to find the influence of various parameters such as normal load, sliding speed, and number of cycles on the tribological behavior of dental materials. The dental wear simulator is a single system that can characterize materials simultaneously at four multiple stations. All stations have the same identical complex movement pattern in two directions (X and Y, mutually orthogonal to the loading direction) over the antagonist disc made of SS-302 (Figure 1).

2.5 Experimental design

The Taguchi method is used to reduce the large number of iterations to a few steps. Instead of having to test all the possible combinations, the Taguchi method tests pairs of combinations and gives the optimum result. This reduces the number of experiments, time, and money. The volumetric wear of nanoalumina and micro-marble dust-filled dental composites were determined using a combination of four factors, each having five levels, and are presented in Table 5. The number of experiments involved was large in number as the effect of each factor on each variable was to be determined. Using the experimental approach, the impact of the four factors and five variables can be studied using the L25 orthogonal array. Using the L25 orthogonal array, the number of iterations evolved was reduced to 25. The S/N ratio for the four factors and five variables was determined by the selected smaller is the better characteristic, as our aim was to reduce the specific wear rate to the minimum. Smaller is the better characteristic was used as Eq. (2).

Figure 1: Block diagram of two-body abrasive dental wear.

Table 5: Levels of the variables used in the experiment.

Control factors	I	II	III	IV	V	Units
Sliding speed	2.5	5	7.5	10	12.5	mm/s
Normal load	20	40	60	80	100	N
Cycles	4000	8000	12,000	16,000	20,000	Cycles
Filler content	0	5	10	15	20	wt. %

$$\frac{S}{N} = -10 \log \frac{1}{n} \left(\sum_{i=1}^n y^2 \right) \quad (2)$$

where, n = the number of observations and y = the observed data, and S/N = the signal-to-noise ratio.

3 Results and discussion

3.1 Effect of hardness on nanoalumina and marble dust-filled dental composite

The hardness of the dental composite filled with different wt. % of nanoalumina and marble dust-filled dental composites are shown in the Figures 2 and 3, respectively. In Figure 2, it was observed that the unfilled dental composite DC-0 indicated a hardness value of 83 Hv, whereas the dental composite filled with 5 wt. %, 10 wt. %, 15 wt. %, and 20 wt. % nanoalumina exhibited hardness values of 94 Hv, 116 Hv, 140 Hv, and 154 Hv, respectively. On the other hand, Figure 3 indicated that the hardness value of the dental composite filled with 5 wt. %, 10 wt. %, 15 wt. %, and 20 wt. % marble dust exhibited hardness values of 94 Hv, 116 Hv, 140 Hv, and 154 Hv, respectively.

Figure 2: Effect of Vickers hardness on the nanoalumina-filled dental composite.

Figure 3: Effect of Vickers hardness on the marble dust-filled dental composite.

and 20 wt. % marble dust was found to be 89 Hv, 102 Hv, 119 Hv, and 134 Hv, respectively. In both the figures, it can be revealed that hardness increased with an increase in filler content. The increase in hardness with the increase in filler content was attributed to the fact that an increase in the hard and brittle ceramic filler such as nanoalumina and marble dust stiffened the elasticity of the matrix material and improved the material resistance to indentations on the surface of the materials. In both Figures 2 and 3, it was also observed that the addition of 5 wt. % of nanoalumina increased the hardness of dental composite by 12%, whereas an addition of 5 wt. % of marble dust increased the hardness of the dental composite by 5 wt. %. More hardness of the nanoalumina-filled composite compared to the marble dust-filled composite was attributed to a stronger binding of nanoalumina with silane compared to marble with silane. It can be also attributed to the fact that a decrease in filler size led to the improvement of the dispersion of the filler in the resin, which resulted in stronger interfacial bonding between the filler and the resin. This result was in agreement with the work by Borges et al. [31] in which it was proposed that nanocomposite and nano-hybrid composite resins possess better Vickers micro-hardness than hybrid and micro-hybrid resins. They further concluded that the incorporation of nano-sized filler particles enhanced the mechanical properties of the dental composites. The increase in surface hardness with the increase in filler loading was in agreement with Kumar et al. [32]. However, the major factor affecting the hardness of the dental composite is the depth of cure of the resins, which depends upon various factors such as source of light polymerization, light intensity, exposure period, and irradiation distance [33, 34].

3.2 Effect of a normal load on the volumetric wear of nanoalumina and marble dust-filled dental composites

The effect of variation of the normal load on the volumetric wear of nanoalumina and marble dust dental composite is depicted in Figures 4 and 5, respectively. The steady state experiments were performed by varying the normal load from 20 N to 100 N in the step of 20 N under constant sliding speed of 5 mm/s and number of cycles of 8000. From both the figures, it can be revealed that an increase in load increased the volumetric wear of dental composites. Simultaneously, the increase in filler content led to a decrease in the volumetric wear of the dental composite.

Figure 4 indicated that for the unfilled composite, an increase in load from 20 N to 100 N increased the volumetric wear by 18.97%, whereas in the case of a 20 wt. % nanoalumina-filled dental composite, an increase in load from 20 N to 100 N increased the volumetric wear by 52.38%. Therefore, an increase in volumetric wear despite an increase in hardness in the 20 wt. % + nanoalumina-filled composite may be attributed to the fact that due to the increase in load, the removed materials acted as a three-body abrasion along with a two-body abrasion.

Figure 5 indicates that in comparison to the 18.97% increase in volumetric wear for the unfilled composite, the 20-wt. % marble dust-filled dental composite indicated an increase in volumetric wear by 76.26% as a result of a load increase from 20 N to 100 N. Again, the increase in wear rate of the composite despite more hardness may be attributed to the weaker interfacial binding force between the matrix and the resin. Later, these removed materials acted as a part of a three-body abrasion. Turssi et al. [35]

Figure 4: Effect of normal load on wear volume of the nanoalumina-filled dental composites.

Figure 5: Effect of normal load on wear volume of the marble dust-filled dental composites.

found that when a normal load was applied, adhesively bonded contact junctions might have been ruptured and detached, generating larger friction coefficients and material loss after many repeated passes of the load.

In this study, less volumetric wear of a nanoalumina-filled dental composite compared to a marble dust-filled composite was in agreement with the work of Say et al. [36]. They suggested that wear resistance was improved with a smaller filler particle size. A more closely packed structure was formed with a smaller particle size, which helped to protect the resin matrix for further erosion [37].

3.3 Effect of sliding speed on volumetric wear of nanoalumina and marble dust-filled dental composites

The effect of variation of sliding speed on the volumetric wear of nanoalumina and marble dust dental composite is depicted in Figures 6 and 7, respectively. The steady state experiments were performed by varying the sliding speed from 2.5 mm/s to 12.5 mm/s in the step of 2.5 mm/s under constant normal load of 40 N and number of cycles of 8000. In both the figures, it can be revealed that an increase in sliding speed increased the volumetric wear of dental composites. It was attributed to the fact that an increase in sliding speed increased the tangential thrust force. Hence, the volumetric wear of the dental composite increased. Increase in sliding speed also increased the friction between two surfaces and their surface roughness. Apart from the normal load and speed, the presence of the medium also affected the wear performance of the dental composite. It has been documented that the pH

Figure 6: Effect of sliding speed on wear volume of the nanoalumina-filled dental composites.

of the working medium affected the degradation rates of the material through catalysis, being more unfavorable to hydrophilic resins [38]. Degradation of resin-based dental materials seems to progress at similar rates in water, artificial saliva, and in neutral to slightly low-pH media [39]. Figures 6 and 7 also indicated that for an unfilled composite, an increase in sliding speed from 2.5 mm/s to 12.5 mm/s increased the volumetric wear by 43.75%, whereas in the case of a 20-wt. % nanoalumina-filled dental composite, it increased the volumetric wear by 23.52%, and the 20-wt. % marble dust-filled dental composite increased the volumetric wear by 29.03%. Hence, it can be seen that apart from the applied load, an increase in sliding speed increased less volumetric wear of the nanoalumina-filled dental composite compared to the marble dust-filled dental composite.

Figure 7: Effect of sliding speed on wear volume of the marble dust-filled dental composites.

3.4 Effect of number of cycles on the volumetric wear of nanoalumina and marble dust-filled dental composites

The effect of variation of the number of cycles on the volumetric wear of nanoalumina and the marble dust dental composite are depicted in Figures 8 and 9, respectively. The steady state experiments were performed by varying the number of cycles from 4000 to 20,000 in the step of 4000 under a constant sliding speed of 5 mm/s and a normal load of 40 N. In both the figures, it can be revealed that an increase in the number of cycles increased the volumetric wear of the dental composites.

The increase in volumetric wear with the increase in the number of cycles was attributed to the fact that increasing the number of cycles increased the sliding distance and time period of the contact between the specimen and

Figure 8: Effect of number of cycles on wear volume of the nanoalumina-filled dental composites.

Figure 9: Effect of number of cycles on wear volume of the marble dust-filled dental composites.

the antagonist disc. These results are in agreement with those reported by other investigators, which found that the volumetric wear in a dental composite increased linearly with the number of cycles, which was proportional to the sliding distance or sliding time [40]. Here, the wear resistant of the nanoalumina-filled composite was more than the marble dust-filled dental composite. It can be attributed to the fact that nano-sized particles occupy the interspacing between the resin matrix, which caused the reduction in erosion of the material. The more fine the particles are in the resin matrix, the more will be the wear resistance. The wear of large particles was more compared to the fine particles as they easily detach.

A study by Manhart et al. [41] showed that the wear of dental composites increased with an increase in the hardness, which is very different from the well-known relationship between wear and hardness. Other studies by Kon et al. [42] and Wonglamsam et al. [43] indicated that a simulated occlusal wear of a composite consisting of porous spherical filler was lower than that of the conventional composites despite the lower flexural strength of the former. These studies also indicated that the mechanical properties and wear of dental composites were not only the effect of filler content but also of filler morphology. Hence, in this regard, the wear of composites consisting of porous spherical filler would be differently influenced compared with that of conventional composites consisting of an irregular-shaped filler.

3.5 Analysis of experimental results by the Taguchi experimental design

The Taguchi experimental approach was applied to determine the dominant factors responsible for the overall reduction in volumetric wear of nanoalumina and marble dust-filled dental composites. The seventh and ninth columns in Table 6 indicate the S/N ratio of the nanoalumina- and marble dust-filled dental composites. The overall mean values for the S/N ratio of the volumetric wear for the nanoalumina- and marble dust-filled dental composites were found to be 50.45 db and 48.61 db, respectively. Also, the sixth and eighth columns in Table 6 indicated the volumetric wear of nanoalumina- and marble dust-filled dental composites as per the Taguchi orthogonal experimental design. It can be seen that the mean volumetric wear of the nanoalumina-filled dental composite (0.00372 mm³) exhibited 9.6% less wear compared to the marble dust-filled dental composite (0.00408 mm³). The results of the Taguchi experimental data for the nanoalumina- and marble dust-filled

Table 6: Experimental design using the Taguchi L_{25} orthogonal array.

S. no.	Filler (wt. %)	Normal load (N)	Sliding speed (m/s)	No. of cycle	ΔV_{AL} (mm ³)	S/N ratio (db)	ΔV_{MD} (mm ³)	S/N ratio (db)
1	0	20	2.5	4000	0.001	60.00	0.002	53.98
2	0	40	5.0	8000	0.003	50.46	0.004	47.96
3	0	60	7.5	12,000	0.004	47.96	0.005	46.02
4	0	80	10.0	16,000	0.005	46.02	0.005	46.02
5	0	100	12.5	20,000	0.006	44.44	0.007	43.10
6	5	20	5.0	12,000	0.005	46.02	0.005	46.02
7	5	40	7.5	16,000	0.006	44.44	0.006	44.44
8	5	60	10.0	20,000	0.003	50.46	0.006	44.44
9	5	80	12.5	4000	0.002	53.98	0.002	53.98
10	5	100	2.5	8000	0.001	60.00	0.001	60.00
11	10	20	7.5	20,000	0.009	40.92	0.007	43.10
12	10	40	10.0	4000	0.008	41.94	0.003	50.46
13	10	60	12.5	8000	0.007	43.10	0.004	47.96
14	10	80	2.5	12,000	0.006	44.44	0.003	50.46
15	10	100	5.0	16,000	0.003	50.46	0.005	46.02
16	15	20	10.0	8000	0.003	50.46	0.004	47.96
17	15	40	12.5	12,000	0.002	53.98	0.005	46.02
18	15	60	2.5	16,000	0.001	60.00	0.004	47.96
19	15	80	5.0	20,000	0.002	53.98	0.005	46.02
20	15	100	7.5	4000	0.001	60.00	0.006	44.44
21	20	20	12.5	16,000	0.001	60.00	0.007	43.10
22	20	40	2.5	20,000	0.002	53.98	0.001	60.00
23	20	60	5.0	4000	0.003	50.46	0.001	47.96
24	20	80	7.5	8000	0.004	47.96	0.001	60.00
25	20	100	10.0	12,000	0.005	46.02	0.004	47.96
					0.00372		0.00408	

 ΔV_{AL} , Volumetric wear of nanoalumina-filled dental composite. ΔV_{MD} , Volumetric wear of marble dust-filled dental composite.

dental composites were analyzed using MINITAB 17 and are presented in Figures 10 and 11, respectively. Figure 10 indicated that the combination of factors should be in the form of A4, B5, C1, D1, and it would give a minimum volumetric wear for the dental composite filled with different wt. % of nanoalumina. It means that the dental composite filled with 15 wt. % nanoalumina at a normal load of 100 N, with sliding speed of 2.5 mm/s and number of cycles of 4000, exhibited a minimum wear rate. On the other hand, Figure 11 indicates that the combination of factors should be A4, B4, C1, D1, and it would give the dental composite filled with different wt. % of marble dust a minimum volumetric wear. It means that the dental composite filled with 20 wt. % of marble dust at a normal load of load of 80 N, with sliding speed of 2.5 mm/s and number of cycles of 4000, exhibited minimum wear rate.

3.6 ANOVA and the effects of factors

A general linear model analysis of variance (ANOVA) was performed to find the significance of factors such as filler

content, normal load, sliding speed, and number of cycles on the volumetric wear of dental composites. ANOVA was performed based on the Taguchi experimental results. Tables 7 and 8 shows the results of the ANOVA with the volumetric wear of nanoalumina- and marble dust-filled dental composites taken from the Taguchi experimental design. This analysis was undertaken for a level of confidence of significance of 5%. The last columns in Tables 7 and 8 indicate the percentage contribution of each factor. The percentage contribution indicates that the main effects are highly significant if the corresponding value of the percentage contribution is low. In Table 7, it can be observed that for the nanoalumina-filled dental composites, the level of significance follows the order filler content ($p=0.055$), sliding speed ($p=0.138$), number of cycles ($p=0.402$), and normal load ($p=0.798$). On the other hand, in Table 8, it can be observed that for the marble dust-filled dental composites, the level of significance follows the order number of cycles ($p=0.037$), filler content ($p=0.043$), sliding speed ($p=0.066$), and normal load ($p=0.453$). Hence, it can be revealed that in the case of a nanoalumina-filled dental composite, the filler content

Figure 10: Effect of control factors on the wear rate of nanoalumina-filled dental composites.

Figure 11: Effect of control factors on the wear rate of marble dust-filled dental composites.

is the most significant factor, whereas in the case of the marble dust-filled dental composite, the number of cycles is the most significant factor influencing volumetric wear of the dental composite. Hence, it can be concluded that

as far as mechanical and wear properties are concerned, nanoalumina should be preferred over marble dust as a filler for the dental composite. Among all the factors, a normal load is the least significant factor.

Table 7: ANOVA table for specific wear volume of nanoalumina-filled dental composite.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Filler content (wt. %)	4	345.46	345.46	86.37	3.67	0.055
Normal load (N)	4	38.35	38.35	9.59	0.41	0.798
Sliding speed (m/s)	4	223.55	223.55	55.89	2.38	0.138
Cycles (rpm)	4	107.60	107.60	26.90	1.14	0.402
Error	8	188.04	188.04	23.50		
Total	24	902.99				

DF, Degree of freedom; Seq SS, sequential sum of square; Adj SS, adjacent sum of square; Adj MS, adjacent sum of mean square; F, variance; P, percentage contribution.

Table 8: ANOVA table for specific wear volume of marble dust-filled dental composite.

Source	DF	Seq SS	Adj SS	Adj MS	F	P
Filler content (wt. %)	4	241.26	241.26	60.31	4.08	0.043
Normal load (N)	4	60.19	60.19	15.05	1.02	0.453
Sliding speed (m/s)	4	200.88	200.88	50.22	3.39	0.066
Cycles (rpm)	4	258.23	258.23	64.56	4.36	0.037
Error	8	118.36	118.36	14.79		
Total	24	878.91				

DF, Degree of freedom; Seq SS, sequential sum of square; Adj SS, adjacent sum of square; Adj MS, adjacent sum of mean square; F, variance; P, percentage contribution.

3.7 Surface morphology

The worn surface morphology of the nanoalumina- and marble dust-filled dental composites studied under steady state conditions using a field emission scanning electron microscope (FE-SEM) is presented in Figures 12 and 13, respectively. FE-SEM was used to characterize the morphology of unworn and worn surfaces of dental composites. Figure 12 indicates the unworn and worn surface morphology of the nanoalumina-filled dental composite under varying conditions of filler content, load, sliding speed, and number of cycles. Figure 12A shows the unworn surface of a 20-wt. % nanoalumina-filled dental composite (DCAL-20). It can be seen that there were a number of uneven and rough faces, voids, and cracks on the surface of the dental composite. Figure 12B and C indicates the effect of varying normal loads on the volumetric wear of the dental composite (DCAL-20). Figure 12B indicates the micro-graph of the surface at 20-N normal load, whereas Figure 12C indicates the micro-graph of a surface at 100-N normal load. It can be observed in both figures that at initial loading, wear track was not visible, but still, there was presence of wear scars and micro-ploughing, which was due to the presence

of an uneven surface before the wear test. However, when the load was maximum at 100 N, wear tracks were visible, and the surface was smooth. Therefore, an increase in load increased the volumetric wear, but wear rate was not significant, which is in agreement with our Taguchi result with the least normal load compared to other factors (Figure 10) and the steady state result (Figure 4).

Figure 12D and E indicates the effect of varying sliding speeds on the volumetric wear of a dental composite (DCAL-20). Figure 12D indicates a micro-graph of a surface at 2.5-mm/s sliding speed, whereas Figure 12E indicates a micro-graph of a surface at a sliding speed of 12.5 mm/s keeping the other factors constant. At a low speed (Figure 12D), the filler particles were exposed and loosened on the surface, and delamination of the upper surface can be seen, but at maximum speed (Figure 12E), the removed particles participated in the three-body abrasion. More wear can be seen at high speed. Figure 12F and G indicates the effect of varying numbers of cycles on the volumetric wear of a dental composite (DCAL-20). Figure 12F indicates a micro-graph of a surface at 4000 number of cycles, whereas Figure 12G indicates a micro-graph of a surface at 20,000 numbers of cycles. At 4000 cycles of the run, the worn surfaces show pores and wear scars on the surface. However, after 20,000 cycles of the run, the surfaces become damaged, and a two-body abrasion can be seen on the surface. Figure 12H and I indicates the effect of varying filler contents on the volumetric wear of a dental composite. Figure 12H indicates a micro-graph of an unfilled dental composite (DC-0), whereas Figure 12I indicates a micro-graph of a 10-wt. % nanoalumina-filled dental composites (DCAL-10). As per Figure 12H, the unfilled composites indicate an interlayer debonding of the matrix creating a fracture on the surface. As per Figure 12I, there was less wear compared to the unfilled dental composite, but still wear scars, small pits, holes were visible.

Figure 13 indicates the unworn and worn surface morphology of a marble dust-filled dental composite (DCMD-20) under different varying conditions of filler content, load, sliding speed, and number of cycles. Figure 13A shows the unworn surface of a 20-wt. % marble dust-filled dental composite (DCMD-20). The fabricated surfaces show a large number of voids, holes, and cracks on the surface. Figure 13B and C indicates the effect of varying normal loads on the volumetric wear of a 20-wt. % marble dust-filled dental composite (DCMD-20). Figure 13B indicates a micro-graph of a surface at 20-N normal load, whereas Figure 13C indicates a micro-graph of a surface at 100-N normal load. It can be observed in both the figures that at 20-N load, wear tracks were visible on the surface, whereas in the same working condition, wear track was

Figure 12: SEM of unworn and worn dental composites under steady state condition. (A) Dental composite DCAL-20 before wear test, (B) DCAL-20 at normal load 20 N (sliding speed at 5 mm/s and number of cycles at 8000), (C) DCAL-20 at normal load 100 N (sliding speed at 5 mm/s and number of cycles at 8000), (D) DCAL-20 at sliding speed 2.5 mm/s (normal load 40 N and number of cycles at 8000), (E) DCAL-20 at number of cycles 4000 (sliding speed 2.5 mm/s and normal load 40 N), (F) DCAL-20 at number of cycles 20,000 (sliding speed 2.5 mm/s and normal load 40 N), (G) DCAL-0 at 40 N (sliding speed at 5 mm/s and number of cycles at 8000), (H) DCAL-5 at 40 N (sliding speed at 5 mm/s and number of cycles at 8000), (I) DCAL-10 at 40 N (sliding speed at 5 mm/s and number of cycles at 8000).

not visible in nanoalumina-filled dental composites (DCAL-20) in Figure 12A. Hence, it can verify our result that the nanoalumina-filled dental composite exhibited better

hardness compared to the marble dust-filled dental composite (DCMD-20). However, when the load was increased up to 100 N (Figure 13C), the worn surface was full of deep

Figure 13: SEM of unworn and worn dental composites under steady state condition. (A) Dental composite DCMD-20 before wear test, (B) DCMD-20 at normal load 20 N (sliding speed at 5 mm/s and number of cycles at 8000), (C) DCMD-20 at normal load 100 N (sliding speed at 5 mm/s and number of cycles at 8000), (D) DCMD-20 at sliding speed 2.5 mm/s (normal load 40 N and number of cycles at 8000), (E) DCMD-20 at sliding speed 12.5 mm/s (normal load 40 N and number of cycles at 8000), (F) DCMD-20 at number of cycles 4000 (sliding speed 2.5 mm/s and normal load 40 N), (G) DCMD-20 at number of cycles 20,000 (sliding speed 2.5 mm/s and normal load 40 N), (H) DCMD-0 at (normal load 40 N, sliding speed at 5 mm/s and number of cycles at 8000), (I) DCMD-10 at 40 N ((normal load 40 N, sliding speed at 5 mm/s and number of cycles at 8000).

grooves, damaged surfaces, wear scars, etc. The delamination of the matrix material was also seen on the surface at a high loading of 100 N.

Figure 13D and E indicates the effect of varying sliding speeds on the volumetric wear of a dental composite (DCMD-20). Figure 13D indicates a micro-graph of a surface

at a 2.5-mm/s sliding speed, whereas Figure 13E indicates a micro-graph of a surface at a sliding speed of 12.5-mm/s. At a low speed at 2.5 mm/s (Figure 13D), the worn surface was smooth, interrupted by numerous cracks extending through the matrix. However, still, the removed materials were exposed on the surfaces along with some voids, which

were left after exfoliation of the filler particles. As the speed was increased up to 12.5 mm/s (Figure 13E), matrix filled debonding can be observed along with some longitudinal cracks perpendicular to the sliding direction. More wear can be seen at high speeds.

Figure 13F and G indicates the effect of varying numbers of cycles on the volumetric wear of dental composites (DCMD-20). Figure 13F indicates a micro-graph of a surface at 4000 number of cycles, whereas Figure 13G indicates a micro-graph of a surface at 20,000 numbers of cycles. At 4000 cycles of run, the worn surfaces were smooth, but existence of voids, pores, and filler material can be seen on the surface. However, after 20,000 cycles of the run, the surfaces become damaged due to micro-ploughing by the two-body abrasion phenomena (Figure 13G).

Figure 13H and I indicates the effect of varying filler contents on the volumetric wear of dental composites. Figure 13H indicates a micro-graph of an unfilled dental composite (DC-0), whereas Figure 13I indicates a micro-graph of 10-wt. % marble dust-filled dental composites (DCMD-10). As per Figure 13H, the unfilled composites indicated more wear compared to the 10-wt. % marble dust-filled dental composites in Figure 13H and the 20-wt. % marble dust-filled composites in Figure 13B. The unfilled dental composite (Figure 13H) shows the presence of longitudinal crack and interlayer debonding of the matrix that led to create a fracture on the surface. As per Figure 13I, wear tracks were visible along with the presence of some wear scars and removed material.

4 Conclusion

The dental composites filled with different weight percentages of nanoalumina and marble dust were successfully fabricated. The Vickers hardness test of the dental composites was performed along with the two-body wear test in artificial saliva medium to assess their mechanical and wear properties. The hardness results indicated that the incorporation of 5 wt. % of nanoalumina increased the hardness of the dental composite by 12%, whereas incorporation of 5 wt. % of marble dust increased the hardness of the dental composite by 7%. The wear result indicated that the experimental composites based on nanoalumina and marble dust have been shown to behave differently in the artificial saliva medium. The nanoalumina-filled dental composites exhibited less volumetric wear compared to the marble dust-filled dental composite. In addition, other factors were found to play an important role in determining the wear performance of dental composites

such as the filler content, applied normal load, sliding speed, and number of cycles.

The steady state condition tests indicated that the volumetric wear of dental composites increased with the increase in normal load and further increased with increasing number of cycles and sliding speed. The Taguchi orthogonal experiment suggested that for the nanoalumina-filled dental composite, the best combination of factors for minimum volumetric wear was the percentage of filler content of 15%, normal load 100 N, sliding speed 2.5 mm/s, and number of cycles 4000. On the other hand, for the marble dust-filled dental composite, the best combination of factors for minimum volumetric wear was percentage of filler content at 20%, normal load 80 N, sliding speed 2.5 mm/s, and number of cycles 4000. Further, it was also concluded that in the Taguchi orthogonal experiments, the mean volumetric wear of the nanoalumina-filled dental composite was 9.6% less than that of the marble dust-filled dental composite in artificial saliva medium.

References

- [1] Yap AU, Wee KE, Teoh SH, Chew CL. *Oper. Dent.* 2001, 26, 349–356.
- [2] Hu X, Marquis PM, Shortall AC. *J. Prosthet. Dent.* 1999, 82, 214–220.
- [3] Yap AU, Teoh SH, Chew CL. *Dent. Mater.* 2002, 18, 149–158.
- [4] Shabani M, Richards LC. *J. Prosthet. Dent.* 2002, 87, 650–656.
- [5] de Gee AJ, Leloup G, Werner A, Vreven J, Davidson CL. *J. Dent. Res.* 1998, 77, 1658–1663.
- [6] de Gee AJ, van Duinen RNB, Werner A, Davidson CL. *J. Dent. Res.* 1996, 75, 1613–1619.
- [7] Yap AU, Teoh SH, Tan KB. *J. Biomed. Mater. Res.* 2000, 53, 547–553.
- [8] de Gee AJ, Pallav P, Davidson CL. *J. Dent. Res.* 1986, 65, 654–658.
- [9] Dickenson GL, Leinfelder KF. *J. Am. Dent. Assoc.* 1993, 124, 68–72.
- [10] Settembrini L, Penugonda B, Fischer E. *J. Clin. Dent.* 1993, 4, 55–60.
- [11] Hotta M, Hirukawa H. *Oper. Dent.* 1994, 19, 42–46.
- [12] Tanoue N, Matsumura H, Atsuta M. *J. Prosthet. Dent.* 2000, 84, 93–97.
- [13] Abenojar J, Martinez MA, Velasco F, Sanchez VP, Martín-Martínez JM. *J. Adhes.* 2009, 85, 216–238.
- [14] Abenojar J, Martinez MA, Velasco F, del Real JC. *J. Adhes. Sci. Technol.* 2011, 25, 2445–2460.
- [15] Abenojar J, Martinez MA, Pantoja M, del Real JC. *J. Adhes.* 2012, 88, 418–434.
- [16] Willmann G. *J. Mater. Process. Technol.* 1996, 56, 168–176.
- [17] Galindo ML, Sendi P, Marinello CP. *J. Prosthet. Dent.* 2011, 106, 23–28.

- [18] Fischer H, Waandich A, Telle R. *Dent. Mater.* 2008, 24, 618–622.
- [19] Stump ASG, Bergmann CP, Vicenzi J, Fetter R, Mundstock KS. *Mater. Des.* 2009, 30, 4348–4359.
- [20] Xia Y, Zhang F, Xie H, Gu N. *J. Dent.* 2008, 36, 450–455.
- [21] Hamburger JT, Opdam NJ, Bronkhorst EM, Kreulen CM, Roeters JJ, Huysmans MC. *J. Adhes. Dent.* 2011, 13, 585–593.
- [22] Deliperi S, Bardwell DN. *J. Am. Dent. Assoc.* 2002, 133, 1387–1398.
- [23] Misra AK, Mathur R, Rao YV, Singh AP, Goel P. *J. Sci. Ind. Res. (India)* 2010, 69, 67–72.
- [24] Baboo R, Khan NH, Kumar A, Tabin RS, Duggal SK. *I.J.C.S.E.* 2011, 1, 827–834.
- [25] Saboya F, Xavier GC, Alexandre J. *Constr. Build. Mater.* 2007, 21, 1950–1960.
- [26] Gangwar S, Patnaik A, Bhat IK. *Silicon* 2017, 9, 249–263.
- [27] Kumar SR, Patnaik A, Bhat IK. *J. Compos. Mater.* 2017, 51, 1997–2008.
- [28] Kelestemur O, Yildiz S. *Sci. Iran. Trans. A: Civ. Eng.* 2016, 23, 896–906.
- [29] Kelestemur O, Anci E, Servet Y, Gokcer B. *Constr. Build. Mater.* 2014, 60, 17–24.
- [30] Kelestemur O, Yildiz S, Gokcer B, Anci E. *Mater. Des.* 2014, 60, 548–555.
- [31] Borges BC, Groninger AI, Soares GP, Santos-Daroz CBD, Ambrosano GMB, Marchi GM, Giorgi MCC, Aguiar FHB. *Dentistry* 2011, 103, 1–5.
- [32] Kumar SR, Patnaik A, Bhat IK. *P. I. Mech. Eng. L-J. Mat.* 2016, 230, 492–503.
- [33] Pires JA, Cvitko E, Denehy GE, Swift EJ. *Quintessence Int.* 1993, 24, 517–521.
- [34] Groninger AIS, Soares GP, Sasaki RT, Ambrosano GMB, Lovadino JR, Aguiar FHB. *Brazilian J. Oral. Sci.* 2011, 10, 189–192.
- [35] Turssi CP, Ferracane JL, Vogel K. *Biomaterials* 2005, 26, 4932–4937.
- [36] Say EC, Civelek A, Nobecourt A, Ersoy M, Guleryuz C. *Oper. Dent.* 2003, 28, 628–634.
- [37] Clelland NL, Pagnotto MP, Kerby RE, Seghi RR. *J. Prothet. Dent.* 2005, 93, 153–157.
- [38] Gopferich, A. *Biomaterials* 1996, 17, 103–114.
- [39] Cilli R, Pereira JC, Prakki A. *J. Dent.* 2012, 40, 1144–1150.
- [40] Callaghan DJ, Vaziri A, Hashemi HN. *Dent. Mater.* 2006, 22, 84–93.
- [41] Manhart J, Kunzelmann KH, Hickel R. *J. Biomed. Mater. Res.* 2000, 53, 353–361.
- [42] Kon M, Kakuta K, Ogura H. *Dent. Mater. J.* 2006, 25, 183–194.
- [43] Wonglamsam A, Kakuta K, Ogura H. *Dent. Mater. J.* 2008, 27, 243–250.