Comparative abrasive wear resistance and surface analysis of dental resin-based materials

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ABSTRACT

Objective: The objective of this study was to assess the surface properties (microhardness and wear resistance) of various composites and compomer materials. In addition, the methodologies used for assessing wear resistance were compared. **Materials and Methods:** This study was conducted using restorative material (Filtek Z250, Filtek Z350, QuiXfil, SureFil SDR, and Dyract XP) to assess wear resistance. A custom-made toothbrush simulator was employed for wear testing. Before and after wear resistance, structural, surface, and physical properties were assessed using various techniques. **Results:** Structural changes and mass loss were observed after treatment, whereas no significant difference in terms of microhardness was observed. The correlation between atomic force microscopy (AFM) and profilometer and between wear resistance and filler volume was highly significant. The correlation between wear resistance and microhardness were insignificant. **Conclusions:** The AFM presented higher precision compared to optical profilometers at a nanoscale level, but both methods can be used in tandem for a more detailed and precise roughness analysis.

Key words: Abrasion, atomic force microscopy, dental materials, Fourier transform infrared, surface roughness

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INTRODUCTION

Wear refers to the progressive loss of material from the surface of teeth or restorative materials as a result of chemical and mechanical processes such as erosion and abrasion.[1] Within the context of the oral cavity, wear is a complex phenomenon. Wear by toothbrushing falls into the category of the three-body wear and is most commonly observed on facial surfaces of teeth and dental restorations.[2] Resistance to abrasive wear is an important property of a dental material. It determines the longevity of the material in clinical service.[3] Surface roughness has a major influence on the esthetic appearance and discoloration of restorations. Microorganisms adhere strongly to rough surfaces, thereby promoting plaque accumulation, caries, and gingival inflammation.[4] Ideal dental restorations should have wear resistance similar to that of tooth.[5] However, till date, wear in composites remains a major concern. The values of average clinical wear on occlusal surfaces of composite restorations approximates to about 29 μm per year for molars and 15 μm for premolars. Substantially higher values are reported for proximal wear.[6]

Considerable improvements have been made in terms of mechanical properties of dental composite resins.[7] Several aspects of the composition and structure of composite resins directly affect and limit wear resistance.[8] There is a paradigm shift in restorative dentistry with the synthesis of new polymeric systems and the introduction of the inorganic nanofillers.[9,10] Manufacturers usually make unsubstantiated claims about the wear resistance of the composite resins. To test manufacturer's claims, an *in vitro* test method was adopted to evaluate mass loss and surface roughness.[11] *In vivo* methodologies are generally very time-consuming and hard to accurately reproduce.[12] Therefore, for the present study, an *in vitro* testing method was employed. The objective of this study was 2-fold, one was to assess the different types of commercially available composites and compomer materials – based on surface properties such as structural changes, microhardness, and corresponding wear resistance. The second was to compare the methodologies used for assessing wear resistance.

MATERIALS AND METHODS

In this study, five different types of commercially available materials microhybrid [Filtek™ Z250 XT, 3M ESPE, Germany], nanocomposites [Filtek™ Z350 XT, 3M ESPE, Germany], packable [QuiXfil, Dentsply, Germany], flowable [Surefull SDR, Dentsply, Germany], and compomer [Dyract® XP, Dentsply, Germany] were selected and their composition is given in Table 1.

Sample preparation

Samples with 10 mm × 2 mm dimension were prepared in Teflon mold for each material. Specimens were prepared in a single insertion and compacted using glass slides on both sides. Samples were thoroughly cured from both sides with 4000 mW/cm^2 irradiance for 60s from each side (Flash Max P4 Ortho, Colorado, USA 1503078). The samples were polished by carefully trimming any excess with a 1200-grit silicon carbide sheets and using an automatic polishing

Table 1: Composition of commercial restorative materials

Product name	Type	Manufacturer	Lot	Fillers	Filler volume (%)	Monomers*
Filtek™ Z250 XT (Z250)	Universal microhybrid composite	3M ESPE, Germany	N703519	Zirconia/silica particle size range 0.01-3.5 μm	50	Bis-GMA, UDMA, Bis-EMA
Filtek™ Z350 XT (Z350)	Nano-composite	3M ESPE, Germany	N660853	Combination of 0.004-0.02 μm nonagglomerated zirconia/silica particles and agglomerated 0.60-1.40 μm clusters	57	Bis-GMA, UDMA TEGDMA, PEGDMA, Bis-EMA
QuiXfil (QFL)	Fast-setting, packable composite	Dentsply, Germany	1503000064	Strontium glass fractions in two sets ranging from particle size 1-4 μm	66	Bis-EMA, UDMA, TCB TEGDMA, TMPTMA
Surefil SDR	Low viscosity, flowable composite	Dentsply, Germany	1503000686	Nano-filled Ba/Si alumino fluorosilicate	45	UDMA, TEGDMA
Dyract® XP (Dyract)	Compomer	Dentsply, Germany	1502000426	Strontium-flouro silicate glass mean filler size 0.8 μm	47	UDMA, TCB

SDR: Smart dentin replacement, Bis-GMA: Bisphenol A glycidylmethacrylate, UDMA: Urethane dimethacrylate, TEGDMA: Triethylene glycol dimethacrylate, Bis-EMA: Ethoxylated bisphenol-A dimethacrylate, PEGDMA: Poly (ethylene glycol) dimethacrylate, TCB: Tetracarboxylic acid-hydroxyethylmethacrylate-ester, TMPTMA: Trimethylolpropane trimethacrylate

machine (Metkon GRIPO 2V Grinder Polisher, Turkey), followed by sonication to remove residue of polishing. Afterward, each specimen was conditioned in distilled water for 7 days at 37°C, according to the conditioning described for abrasive wear test IS0/TR 14569. The samples were then air-dried for an hour. Readings for all tests were recorded before and following the abrasive wear test.

Fourier-transform infrared spectroscopy

To find the structural changes, spectroscopic analysis of all samples was conducted before and after treatment. For each sample before and after treatment, 10 spectra were taken to find out the spectral difference. Thermo Nicolet 6700 (USA) was used with attenuated total reflectance as accessory. The resolution was 8 cm^{-1} with 256 scan number. The spectral range was 4000–600 cm^{-1}. OMNIC software (Thermo Fisher Scientific wissenschaftliche Geräte GmbH, Austria)was used to analyze the spectra.

Principal component and cluster analysis

Chemometric methods were used to quantify the spectral differences of various composite groups, i.e., untreated Dyract, untreated QFL, untreated SDR (US), untreated Z250 (U2), untreated Z350 (U3), treated Dyract, treated QFL, treated SDR (TS), treated Z250 (T2), and treated Z350 (T3). These methods were performed using Unscrambler X 10.2 software, purchased from Camo software (Oslo, Norway). Preprocessing comprised of baseline correction and unit vector normalization. Cluster analysis (CA) was performed over complete spectral range by Ward's method using squared Euclidean distance.

Microhardness testing

For a comparison of selected materials before and after simulated toothbrushing, microhardness was analyzed in terms of Vickers hardness number. Using a 200 g load with 10s dwell time (Microhardness tester, WOLPERT, 401MVD EQPT 0002, Germany). Six samples from each group were used and each specimen was indented three times at three different points, and then the mean reading was recorded.

Weight analysis

Before the abrasive test, samples were weighed using an analytical electronic balance (Sartorius AG Gottingen BP 110 S, Germany) with accuracy up to 0.1 mg. In this way, initial mass (M_1), for each sample was obtained. Following the abrasive wear test, the samples were carefully removed, rinsed in tap water, and placed in an ultrasonic water bath (Cole-Parmer, Vernon Hills, Illinois, USA) for 1 min. The samples were then individually removed, air-dried, and weighed. In this way, final mass (M_2) was obtained for each sample and mass loss (%) was reported for each material post abrasion using following equation:

$$W\% = ([M_2 - M_1]/M_1) \times 100\% \quad (1)$$

Surface roughness analysis

Initial surface roughness was assessed using a noncontact mode 2D Profilometer (PS-50 Nanovea, Russia) and using a 3D-Atomic Force Microscope (AFM SPM-9500J3, Shimadzu Corp, Japan), operating in tapping mode. Micrographs were obtained at different scan areas measuring 20 μm × 20 μm using AFM software (SPM-0ffline Shimadzu Corp. Japan). The roughness average, Ra, is the most widely used one-dimensional (1D) roughness parameter, and it denotes the arithmetic mean of the absolute values of the collected roughness data points. Ra_i (initial values) were taken and means were obtained. Surface roughness (Ra_f) was measured after the abrasion wear test in the same way as for initial values, except that the tracing arm of profilometer and tip of AFM were positioned in such a way that the tracing direction was perpendicular to the direction of tooth brushing action. 3D images were reported in area selection of 10 μm × 10 μm.

Abrasive wear test

For the abrasive wear test, a custom-made toothbrush simulator was constructed in accordance with ISO11609: 2010, equipped with six stations of replaceable brush heads (Oral B Flat end). Tooth brushing load of 1.5 N was set. To mimic the original condition of toothpaste (Colgate-Palmolive, Dublin, Ireland), slurry was made with distilled water in the proportion 1:2. Resin-based samples were mounted in impression compound and placed in metallic stations. Toothbrushing was accomplished with horizontal movements of toothbrush and travelled a course of 4.2 cm. Time duration was kept 100 min amounting to about 12,250 strokes was set. Toothbrushing time of 1.3 years was simulated. With these parameters, a minimum weight loss of 2 mg by reference material, as described in IS0/TR 14569, was achieved. The slurry and brush heads were replaced for each sample.

Data analysis

The data were submitted to the analysis of variance (ANOVA) and *post hoc* Tuckey's Test, using

IBM SPSS statistics version 21, Boston, Massachusetts, USA. The Pearson's test was used to verify the correlation between Roughness averages reported by AFM and Profilometer. In addition, the correlation of roughness alteration with filler volume and microhardness was also determined.

RESULTS

Fourier-transform infrared spectroscopy

Fourier-transform infrared (FTIR) spectra of samples (Z250, Z350, QFL, SDR, and Dyract) were collected before and after treatment as shown in Figure 1a-e.

The spectra of untreated samples showed C = O stretching vibration peaks at 1710–1715 cm^{-1}, peak at 1653 cm^{-1} and 1633 cm^{-1} attributed to C-C symmetric stretching appeared in all samples except SDR as aromatic group is not present in this composite. Peak at 1510 cm^{-1} corresponded to N-H bending vibrations of urethane-based resins. C-H bend was observed at 1462 cm^{-1}. The overlapping peaks at around 1250–900 cm^{-1} were due to asymmetric stretching vibration of C-O-C of monomer structure. Another sharp peak at 771 cm^{-1} was due to C-H vibrations. After treatment, changes in peak intensities were observed for all samples specifically at C = O and N-H groups.

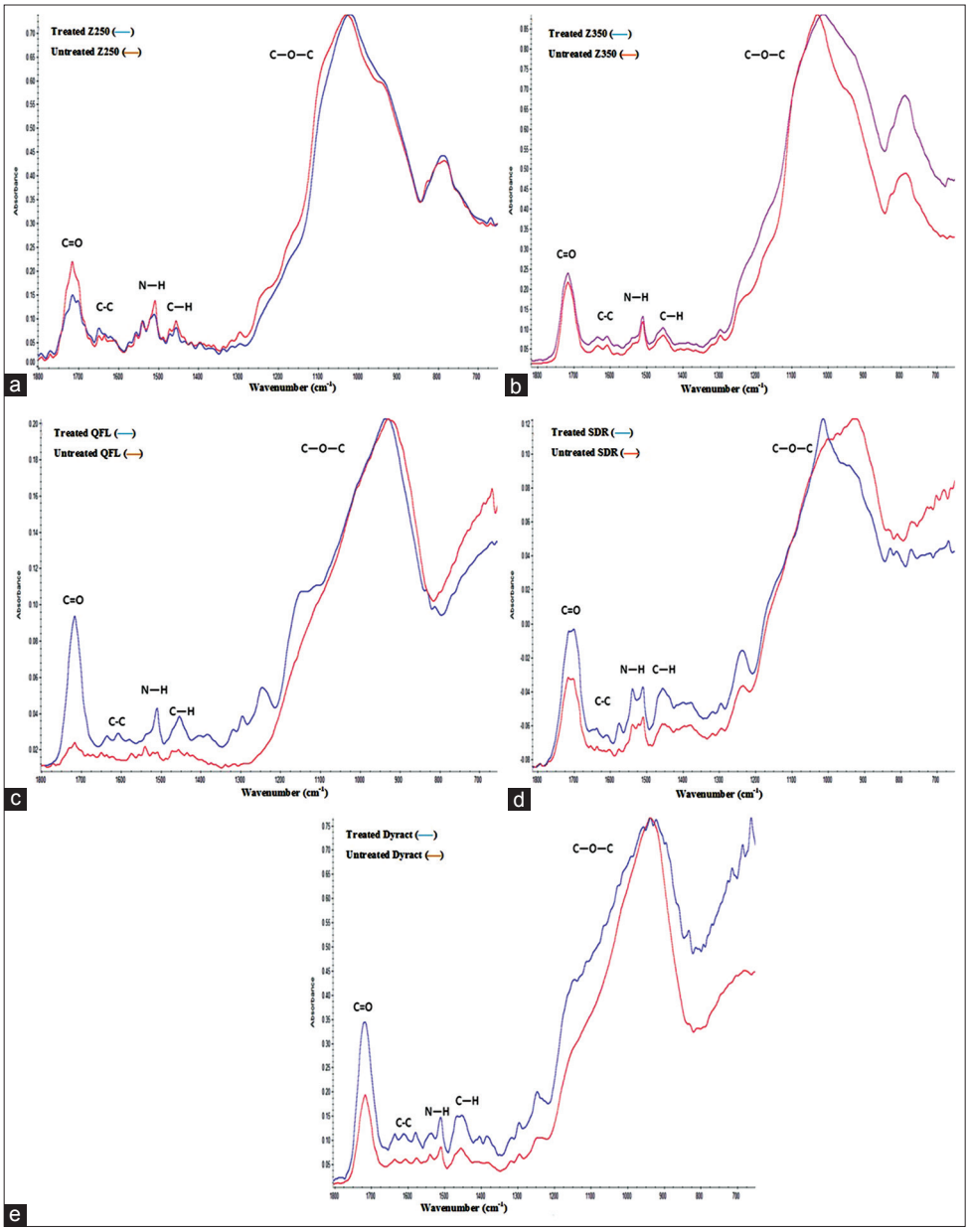

Figure 1: Comparative Fourier-transform infrared spectra of treated and untreated restorative materials; (a) Z250, (b) Z350, (c) QFL, (d) SDR, and (e) Dyract

Principal component analysis

Principal component analysis (PCA) was performed and a comparison (spectral range; 700–3100 cm^{-1}) was conducted between various treated and untreated dental composites as shown in Figure 2. Different comparative spectral ranges, 1660–1760 cm^{-1}, 1590–1650 cm^{-1}, 1420–1470 cm^{-1}, and 820–1220 cm^{-1} are given in Figure 3a-d, respectively. Complete spectral range displayed favorable separation of treated and untreated dental composites; PC1 separated all composites from TS with 97% variance, whereas the remaining 2% was observed in PC2 and 1% in PC3 (figure not shown). All samples have shown distinct cluster formations to be recognizable as one group; however, US and T3 have demonstrated scattered formations hence suggesting inner group variability [Figure 2].

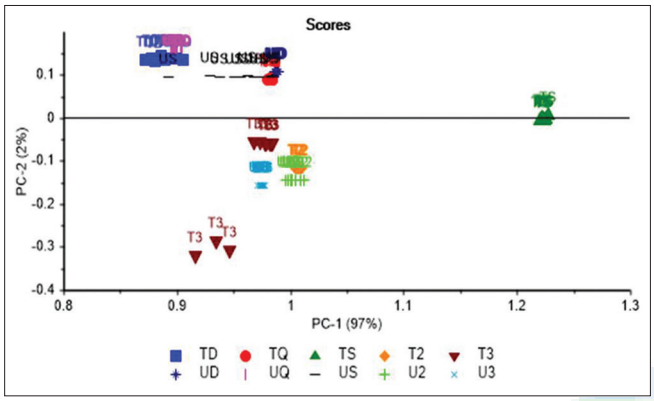

Figure 2: Principal component analysis of all treated and untreated composites over the complete spectral range

Similar trends were observed at 1660–1760 cm^{-1} region; however, the variance observed by PC1 was improved to 98% and the remaining 2% was detected by PC2. Within the treated and untreated composites, the sample groups can be well discriminated using PCA, whereas US and T3 showed variations within their respective groups [Figure 3].

By comparison, differences within the treated and untreated groups appeared to be much greater in the 1590–1650 cm^{-1} region, and the scores plot for this region showed good separation between all groups, i.e., 99% for PC1. While the clusters were more widely spread, loading plots for PC1 and PC2 (data not shown) suggested the contrast in the peaks at this region to be a major influence. Within the treated and untreated samples at 1420–1470 cm^{-1} region, the sample groups can be well discriminated using PCA. PC1 and PC2, accounting for 100% of the variance, discriminated all samples on the basis of

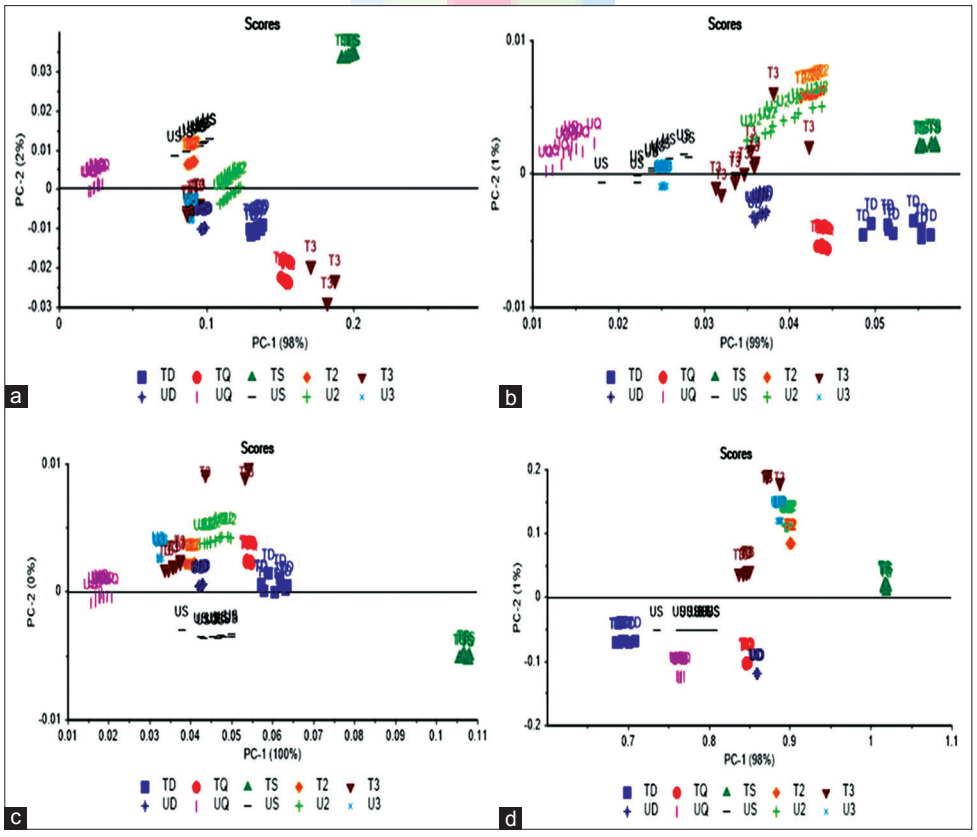

Figure 3: Principal component analysis of all treated and untreated composites over (a) 1660–1760 cm^{-1}, (b) 1590–1650 cm^{-1}, (c) 1420–1470 cm^{-1}, and (d) 820–1220 cm^{-1} region

their chemical content. The distribution of individual composite type in relation to the variance explained by PC1 produced clear and separated clusters for each of these samples. PCA between treated and untreated samples at 820–1220 cm^{-1} region showed that the variance explained by PC2 separated T2, U2, T3, and U3 from all other composite types, whereas PC1 distributed TS away from the others. Every composite type formed a separate cluster with the exception of US and T3 where PC1 demonstrated 98% variance and PC2 2% variance.

Cluster analysis

CA was performed over the complete spectral range (700–3100 cm^{-1}). Figure 4a showed the dendrogram of classification results for a dataset comprising of spectra collected from all untreated dental composites. Two distinct branches are formed where Z250 and Z350 were grouped together, whereas SDR, QFL, and Dyract have clustered separately. Altogether, each of the different composites formed well-defined clusters; however, SDR represented maximum relative distance hence suggesting most inner group variability. Figure 4b showed the dendrogram of classification results for a dataset comprising of spectra collected from all treated dental composites.

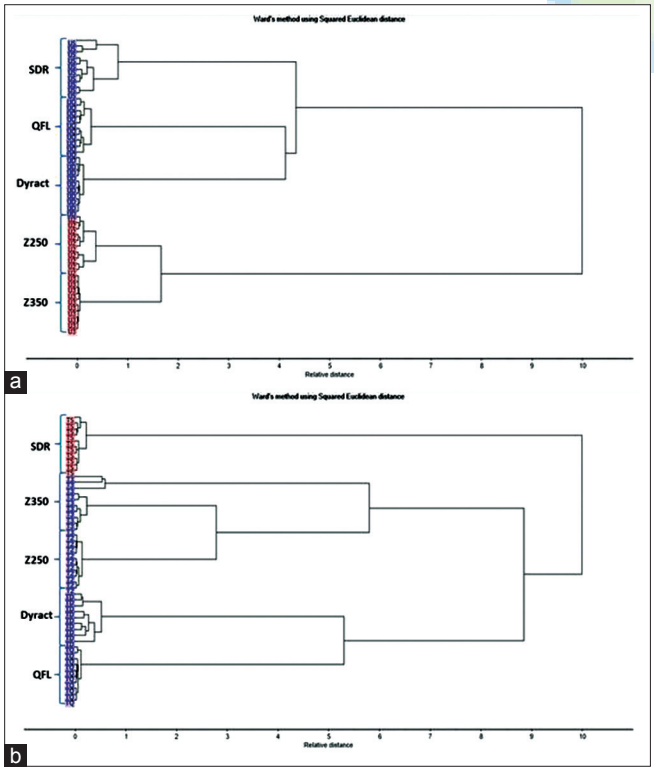

Figure 4: Component analysis of all (a) untreated and (b) treated samples over the complete spectral range

Two distinct branches are formed where SDR was clearly separated from the remaining treated composites. All sub-branches purely contained specific composite types with no one mixing with the other hence complementing the sensitivity of the technique. Dyract and Z350 demonstrated inner group variability of treated composites on the basis of maximum relative distance as compared to the rest.

Hardness testing

Comparison of microhardness among treated and untreated composite materials is tabulated in Table 2. Dyract and QFL showed highest and lowest change, respectively, in microhardness compared to all the composite materials tested. A statistically significant change in microhardness was found between the groups after simulated toothbrushing ($P > 0.05$) by one-way ANOVA. *Post hoc* Tukey's test showed a significant difference between change in hardness of Dyract and QFL as well as Z350. There was also a significant difference between the results of QFL and SDR ($P > 0.05$). In the context of wear, all tested materials suffered significant mass loss ($P < 0.05$). Percentage mass loss of each material is graphically depicted in Figure 5. The maximum mass loss was observed in Z250 while Dyract showed the lowest mass loss. The mass loss observed in the case of SDR was significantly higher than the other tested composite materials ($P < 0.01$).

Surface roughness

Initially, all tested materials presented relatively low values of surface roughness, as polishing of all samples was performed before the abrasion test. However, as expected toothbrush abrasion caused visible nanoscale alterations on the surface of all samples, varying in extent, according to material as illustrated by 3D pre- and postabrasion test

Figure 5: Average weight loss percentages of samples after treatment

images obtained with the help of AFM [Figure 6a-e]. One-way ANOVA indicated that there was significant difference in the R_a values between the groups ($P < 0.05$) using both AFM and optical profilometer.

The initial and final surface roughness values obtained with AFM and profilometer are illustrated graphically in Figure 7a and b, respectively. Following toothbrush abrasion, all tested materials presented a statistically significant increase in roughness values ($P < 0.05$). Z25O presented with the highest Ra_f value after abrasion test and also depicted the highest roughness alteration [Figure 7a and b].

Dyract suffered highest mass loss the smoothest surface and the lowest Ra_f value. Two-tailed Pearson's correlation was used to verify the correlation between AFM and optical profilometer, and it was found to be highly significant ($P < 0.01$). In addition, the correlation of roughness alteration (rate of wear) and microhardness was found to be significant with filler volume ($P < 0.05$). The correlation between change in microhardness and surface roughness alterations of the tested materials with both methodologies, however, was not significant ($P > 0.01$).

Table 2: Comparison of microhardness among the tested materials

Material	Mean microhardness (VHN)
Dyract XP	103.1±17.04
Filtek™ Z250	92.93±11.21
Filtek™ Z350	109.06±22.86
QuiXfil	100.3±13.73
Surefil (SDR)	84.46±7.75

VHN: Vickers hardness number, SDR: Smart dentin replacement

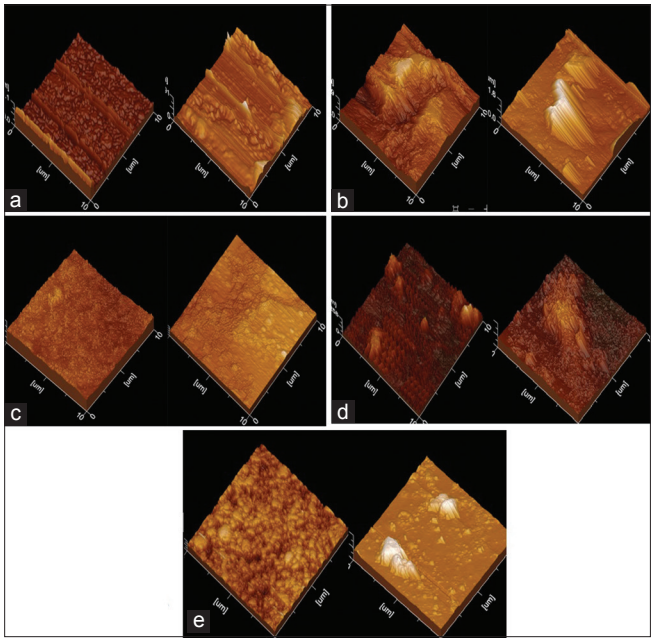

Figure 6: Three-dimensional atomic force microscopy images of restorative materials; (a) Z250, (b) Z350, (c) QFL, (d) SDR, and (e) Dyract, before (left) and after (right) treatment

DISCUSSION

In recent years, considerable improvements in the properties of dental composites have been made; however, wear of composite still remains a concern.[6] In this aspect, the surface properties of restorative material play a major role in the long clinical life of restoration. In the oral cavity, wear is reflected by tearing away of organic matrix, exposure of inorganic content, and loss of smaller filler particles due to chewing and due to toothbrushing in our daily life.[13] This surface roughness results in the loss of esthetics and also leads to an increase in accumulation of dental plaque and lodging of food particles, which coupled with bacterial adhesion, subsequently results in the destruction of restoration.[14]

In this study, along with the comparison of the wear of latest available materials, different available methodologies were used to analyze wear and correlation among these methodologies was found. For composite restorative materials, composition and filler morphology play a major role in its resistance

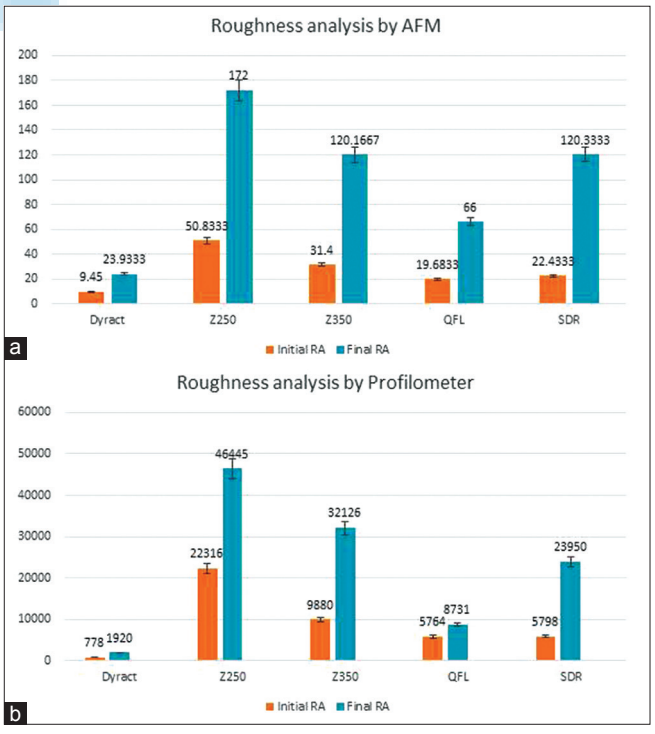

Figure 7: Surface roughness values of restorative materials using (a) atomic force microscopy and (b) optical profilometer

against wear.[15] The various materials selected for this study varied with respect to aforementioned variables.

Chemometric aids have demonstrated excellent sensitivity and specificity over the past two decades in various research areas from biological tissues to synthetic materials. These algorithms eliminate the chances of interobserver variability and provide true reflection of the data provided. PCA was chosen as a tool to investigate the spread of both treated and untreated dental composite groups and inner-group variation with various spectral regions selected based on the initial differences observed in the data. Although visual evaluation of the spectra showed similar peaks, each model presented as a separate cluster in PCA and CA. The clear separation between and within different dental composites suggested the substantial differences in the biochemical composition. Distinct characteristics were observed for SDR samples both in terms of inner-group variability as well as between groups. These results were consistent with the findings of FTIR data where the absence of C-C symmetric stretching was observed due to lack of aromatic groups. CA formed on the basis of molecular differences between the dataset showed separate branches in the dendrogram for each composite type. Both the treated and untreated revealed further discrete subsets representing each of the five composite types, and this discrimination suggested a high sensitivity of the technique. CA also supported the findings related to the absence of aromatic groups by classifying SDR in a separate branch of the dendrogram both in treated and untreated states.

The general concept is that, by measuring microhardness of material, a better understanding of the resistance of material against wear can be obtained. The measurement of microhardness theoretically implies that a hard surface will suffer less abrasive wear than a soft surface if other factors remain constant. However, previous studies showed controversial results, where it was reported that no significant interactions between hardness and wear.[4,16,17] On the other hand, a significant relationship between hardness and wear has also been reported by Wang *et al*.[18]

Before toothbrushing simulation, SDR showed minimum microhardness values which were due to low filler content as compared to other composite materials. The results were in agreement with previous studies.[16,17] Z350 (nanocomposite) showed high hardness value compared to Z250 (microhybrid composite) which might be due to higher filler loading and higher surface area of filler particles, which have tendency to improve the interfacial linkage between resin-fillers. Postabrasion, Dyract and SDR presented with the greatest reduction in surface hardness which can be attributed to lower filler content compared to other materials.[17] For Dyract, this might be due to the surface dissolution on contact with water, whereas Z350 and QFL presented with an increase in surface hardness after toothbrushing. This might be attributed to the surface deposition of dentifrice slurry on the surface of these materials. No change was observed in the surface hardness of Z250. However, the overall results of the present study did not find any significant correlation between changes in microhardness and wear resistance ($P > 0.05$).

Roughness average (Ra) is a well-accepted comparative feature, which quantifies surface texture by means of randomized readings of amplitude.[19] In most previous studies, Ra_f was interpreted as the only predictor of roughness and change in surface roughness which corresponds directly to rate of wear is usually not mentioned. A composite material with a high Ra_i will also have a high Ra_f. The previous studies lacked in reporting the change in surface roughness and compared the two equipment (profilometer and AFM) on the basis of roughness values (Ra_f) thus acting as a confounder.

The scale and resolution of the results generated by the profilometer and the AFM are not comparable. However, the results for each of the five groups being tested showed a similarity in trends in mutual comparison when it came to increasing R_a for both testing techniques.[4,13] The present study showed a significant correlation between the two contrary to a previous study which showed no correlation between the Ra values obtained using both techniques.[12,16]

Among composite material, QFL showed best results in terms of minimum change in roughness and final roughness average. Even though QFL showed the smoothest surface, but unlike compomers, it demonstrated lower mass loss. The mass loss was only more than SDR. The better wear resistance can be due to the higher filler volume along with better bonding between the filler and matrix component. The presence of triethylene glycol dimethacrylate (TEGDMA) can be another reason of better wear resistance as it enhances the filler-matrix interaction and improves polymerization which reduces the effect of water sorption.[20,21] TEGDMA has a polyether backbone that increase its flexibility,[22] and this may allow better molecular interaction and

hence better polymerization. This results in increased degree of conversion reducing sorption and making the structure stiffer.

The minimum mass loss was recorded for the SDR, even though it presented with a greater change in roughness compared to Dyract and QFL. The minimum mass loss might be attributed to the presence of smaller particles and reduced interparticle spacing resulting in even distribution that favors the matrix against tearing completely. Greater mass loss in the case of Dyract and SDR complements the reduction in surface microhardness of these materials. However, for roughness changes, according to previous studies, higher filler-loading resulted in greater wear resistance.[23] The presence of lower filler content of flowable composite than packable composites explained the higher change in roughness. Not only the filler volume but also the filler-particle size affects the wear resistance.[24] Turssi *et al*.[25] suggested that the presence of large particles theoretically cause greater abrasion. Increase in filler-particle size, causes an increase in the coefficient of friction, and stress spreads readily from the filler particles to the resin matrix, resulting in greater wear. In addition, wear affects the surface properties of materials such as hardness and elastic modulus.[26] This was reflected in the comparison of particle sizes of microhybrid material Z250 and nanocomposites Z350. Due to higher surface area and surface energy, nanosized particles improve the performance of resin composites.[27,28]

On comparing the mass loss of Z350 with Z250, the former showed significantly lower than latter ($P < 0.01$). Z350 presented a smoother surface when compared with the Z250. This was due to the more homogenous distribution and greater volume of filler content of nanocomposites.[29] Overall, Z250 presented with the greatest mass loss and roughest surface after toothbrush abrasion test. This was somewhat expected, as when any of the hybrid materials are subjected to abrasion, the resin between and around the heterogeneous filler-particle distribution is lost, leading to protruding filler particles. Over time, the entire filler particles are plucked out creating bumps and craters and a highly roughened surface.[30] There are a few limitations of this *in vitro* study; the wear of materials was analyzed in the laboratory set up where oral environment could not be simulated. This study was limited to abrasive wear. Clinically, toothbrushing may affect the rate of abrasive wear depending on hardness of bristles and abrasiveness of dentifrices. The variations and complexity of oral environment may affect the wear behaviors and clinical performance of restorative materials.

CONCLUSIONS

The structure and composition of composites and compomer materials, in particular, the matrix characteristics, type of filler, and filler-particle size greatly affect the wear resistance. FTIR along with PCA/CA confirmed structural changes and revealed information about chemical groups prone to bring change in materials properties. Greatest mass loss was reported by Z250 while SDR suffered the minimum mass loss. The smoothest surface was demonstrated by the Dyract while the roughest surface was that of Z250. AFM and Optical profilometer can be used in tandem for roughness analysis and correlation between these techniques are highly significant, where AFM offered a higher precision at a nanoscale level.

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Conflicts of interest

There are no conflicts of interest.

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