

## Surface roughness analysis of nanocomposite discs polished with two conventional systems and a novel mesoporous nanoabrasive using atomic force microscopy

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### Abstract:

**Background:** The major reason for failure of composite restorations has been attributed to secondary caries in various randomized controlled trials. Proper finishing and polishing of the restoration can reduce plaque accumulation and formation of secondary caries. Nano abrasives are more effective in reducing the surface roughness of nanocomposites than the conventional micron sized abrasive polishing systems. **Aim:** To evaluate the surface roughness of nanocomposite resin discs using Atomic Force Microscope (AFM) after polishing using two different commercial polishing kits and a novel mesoporous nanosilica abrasive. **Materials and Methods:** Nanosilica was prepared by a sol-gel method to obtain a mesoporous structure with a 6mm pore arrangement. Sixty nanocomposite resin discs of Filtek Z250 XT (3M ESPE Dental Products, St. Paul, MN, USA) were prepared and divided into 4 groups. Group1- unpolished, Group2- polished with Sof-Lex system (3M ESPE Dental Products, St. Paul MN, USA), Group3- polished with Super-Snap (Shofu Inc., Kyoto, Japan) and Group4- polished with porous nanosilica abrasive slurry. Average surface roughness values (Ra) were measured using an Atomic Force Microscopy (AFM). **Results:** Group 1 (unpolished) showed the highest Ra values followed by group 3 (Super-snap) and group 2 (Sof-lex). Group 4 (porous nanosilica) showed the smoothest surface in AFM after polishing. Statistical analysis was done using one- way ANOVA and Tukey's post hoc tests which demonstrated a highly significant difference ( $p < .001$ ) between the mean values of all the 4 groups. **Conclusion:** Within the limitations of this in vitro study, it was concluded that the smoothest surface with least Ra values were produced by porous nanosilica abrasive slurry when compared with the commercially available micro polishing systems- Sof-lex and Super-Snap.

**Key Words:** Atomic Force Microscope, Average surface roughness, Mesoporous nanosilica, nanocomposite,

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### I. Introduction

Secondary caries remains the most important reason for failure of composite restoration even though modifications were done in every aspect of its composition. The recent nanocomposites are superior to the early ones in terms of physical and mechanical properties. It has got good handling properties, greater polishing ability, high stain resistance, good color stability and a low wear rates<sup>1</sup>. The final esthetic appearance depends on the artistic ability of the clinician, contouring and shaping & the finishing and polishing of the restoration. Effective finishing and polishing also provides acceptable oral health of soft tissues and the marginal integrity of the restorative- periodontal interfacereducing plaque accumulation and secondary caries formation<sup>1</sup>. Conventional finishing and polishing systems are coated with the micron-sized silica and aluminium-oxide particles. Composites with nano fillers polished with micro abrasives causes roughness and plaque accumulation leading to secondary caries.

According to the concept of chemical-mechanical planarization (CMP), nano abrasives are able to produce a smoother and finer surface<sup>2</sup>. Various types of nanosilica abrasive slurries have been used in CMP which have been traditionally used for polishing the semiconductors, computer hard discs etc. to a nano level. Colloidal silica nanoparticle has been used for polishing the tooth in order to reduce the bacterial adhesion for preventing dental caries<sup>3</sup>. These nano abrasives are very stable, have good biocompatibility, easy method of preparation and a very low cost. Porous nanosilica abrasives have been tried in CMP which produces fewer scratches and lower surface topographical variations with efficient Material Removal Rate (MRR). Hence, this

study was conducted to assess the efficiency of porous nanosilica abrasive in polishing nanocomposite compared with conventional micron- sized aluminium oxide polishing discs.

## II. Material And Methods

**Specimen preparation:** Sixty nanocomposite discs of Filtek Z250 XT (3M ESPE Dental Products, St. Paul, MN, USA) were prepared using an aluminium mold of 10x2mm and then fixed to a cover slip using water insoluble glue. The samples were standardized before polishing using a surface profilometer (Surtronic 3+, Taylor Hobson Ltd, Leicester, England). The surface roughness was kept at a cut- off value of 0.8mm and the traversing distance of stylus was 6mm. The radius of the tracing diamond tip was 5µm and the measuring force and speed were 1mm/sec.

The samples were then randomly divided into four groups of 15 each, (n=15).

**Group 1** Unpolished nanocomposite resin discs.

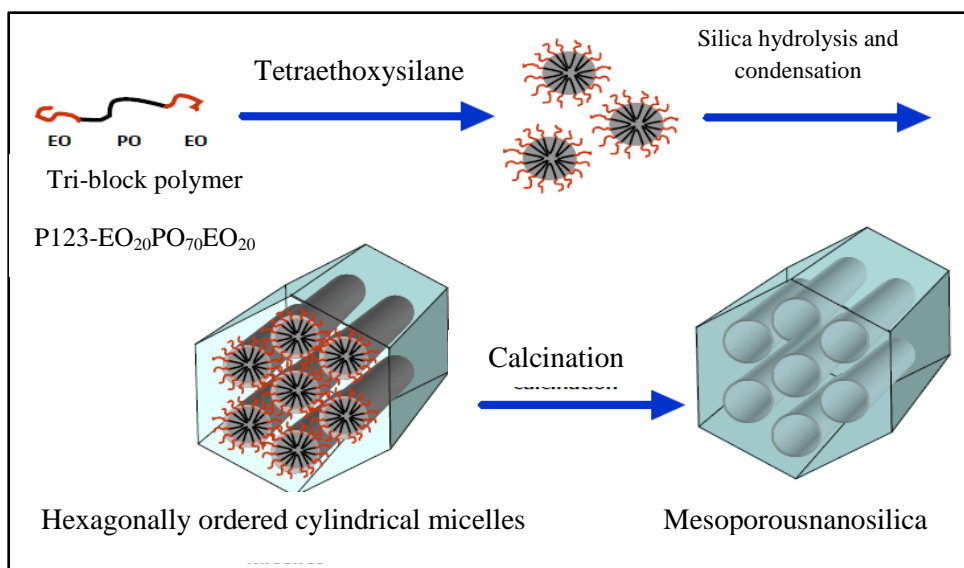
**Group 2** → Polishing with **Sof-Lex** discs (3M ESPE Dental Products, St.Paul MN, USA).

**Group 3** → Polishing with **Super-Snap** (Shofu Inc., Kyoto, Japan).

**Group 4** → Polishing with **porous nanosilica** abrasive slurry.

**Synthesis of mesoporousnanosilica abrasive:** Mesoporousnanosilica with a typical P6mm pore arrangement was synthesized by sol- gel method<sup>4</sup>. 2.3 g of the amphiphilic Tri-Block Polymer Pluronic P123 (Poly(ethylene glycol)-block-poly (propylene glycol)-block-poly (ethylene glycol), Mav=5800, EO20PO70EO20, Sigma Aldrich Corporation, USA acts as the templating agent or structure directing agent to synthesize large- pore mesoporous materials. The pluronics were dissolved in 15 ml of ethanol and stirred for 2 h. Then 4.16 g of the silica source Tetraethoxysilane(Sigma Aldrich Corporation, USA)was added to the above mixture and stirred for 1 hr. The resulting solution underwent solvent evaporation at room temperature for 2 days to get a rigid gel which was then dried at 80 °C for 12 h to remove the residual ethanol. Finally, the samples were calcined at 550 °C in air for 5 h with a heating rate of 1 °C/min to remove the surfactant. During the process of silica hydrolysis and condensation, the shape of the spherical micelles changes to rod-like. After silica condensation, the organic template is removed by calcination, thus creating the large mesopores. This material exhibits microporosity originating from the corona micellar chains which are burned upon calcination. The average particle size of the hence synthesized porous silica nanocomposite abrasive was measured using Scanning Electron Microscopy. The size obtained was 70 nm.

A schematic representation of the synthesis is given below:



**Polishing procedure:** The GROUP 2 samples were polished sequentially using the Sof- lex system with medium discs of 40 µm size coated aluminium oxide particles, fine discs of 24 µm particle size and ultrafine discs with 7 µm size. The GROUP 3 samples were polished using Super Snap with medium discs of aluminium oxide particles (35 µm size), fine discs (20 µm size) and superfine (8 µm size). All the discs were attached to its respective mandrel mounted on to the slow speed contra- angle hand piece (NSK, Japan) rotating at a speed of 20,000 rpm. The whole procedure was carried out by a single operator with a light pressure and brushing strokes for 20 seconds per disc. A new series of discs were used for each specimen and the samples were rinsed in running water to remove the debris.

The group 4 samples were first smoothed with finishing diamond burs (TF- 12EF) using a light pressure. The porous nanosilica abrasive slurry was then applied and polished with the help of a conical rubber cup for 20 seconds with light pressure in a circular motion. After polishing the discs were rinsed in running water to remove the debris.

**AFM analysis:** After completion of the polishing procedure all the samples were imaged using the Atomic Force Microscopy. AFM is a very high resolution type of scanning probe microscopy with resolution fraction of a nanometer. The imaging technique is based on the detection of deflective forces between the silicon cantilever with a sharp tip and sample surface. In this study, AFM (Park Systems Corporation, Suwon, Korea) operating in non-contact mode was used. A  $10\mu\text{m} \times 10\mu\text{m}$  area for imaging was randomly selected with the ‘V’ shaped silicon cantilever in 1 Hz with  $256 \times 256$  pixel resolution. The mean surface roughness (Ra) value calculations were done with the AFM in built- Park XEI 100 Version- 1.8.3 software.

**Statistical analysis:** Data was analyzed using SPSS version 20 (SPSS Inc., Chicago, IL). Analysis of variance (ANOVA) and Post hoc test – Tukey HSD tests were employed to ascertain the significance of differences between mean values of the two groups. The  $P < 0.05$  was considered as the level of significance.

### III. Result

The surface roughness (Ra) values in nm obtained after AFM analysis

**TABLE – 1:** The surface roughness (Ra) values in nm obtained after AFM analysis

SPECIMEN No.	GROUP 1 UNPOLISHED	GROUP 2 SOFLEX	GROUP 3 SUPERSNAP	GROUP 4 NANOSILICA
1	38.569	17.956	23.458	7.953
2	40.933	20.475	22.490	9.903
3	38.964	17.978	24.921	7.361
4	40.536	20.743	24.435	6.335
5	42.000	21.978	23.131	6.842
6	42.469	20.590	24.176	7.355
7	38.134	16.549	23.864	7.913
8	38.676	16.433	22.908	6.033
9	39.649	17.546	23.822	6.527
10	39.479	16.435	24.347	7.414
11	41.361	19.231	23.154	7.193
12	40.897	20.683	24.186	6.200
13	40.009	16.567	24.178	7.413
14	42.546	17.654	23.190	7.001
15	39.698	18.134	24.267	7.054

**TABLE-2:** The mean and standard deviation of Ra values of all the 4 groups

Group	Mean	Std. Deviation	Std. Error
Group 1	40.262000	1.4202273	0.3667011
Group 2	18.597333	1.8696733	0.4827476
Group 3	23.769333	0.6806244	0.1757365
Group 4	7.233133	0.9357351	0.2416058

**TABLE-3:** ANOVA analysis after polishing

AFM values	Sum of Squares	df	Mean Square	F	Sig.
Between Groups	8481.047	3	2827.016	1650.433	.000
Within Groups	95.922	56	1.713		
Total	8576.969	59			

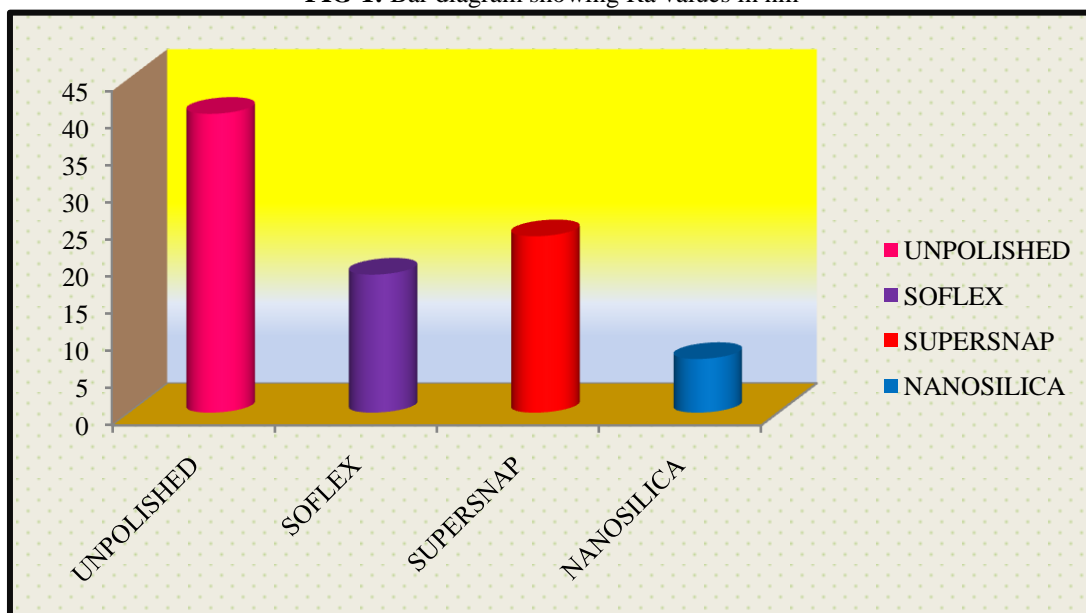
**TABLE-4:** Post Hoc Tests after polishing- Multiple Comparisons

Tukey HSD						
I GROUPS	J GROUPS	Mean Difference (I-J)	Std. Error	Sig.	95% Confidence Interval	
					Lower Bound	Upper Bound
GROUP1	2	21.6646667*	.4778973	.000	20.399249	22.930085
	3	16.4926667*	.4778973	.000	15.227249	17.758085

	4	33.0288667*	.4778973	.000	31.763449	34.294285
GROUP2	1	-21.6646667*	.4778973	.000	-22.930085	-20.399249
	3	-5.1720000*	.4778973	.000	-6.437418	-3.906582
	4	11.3642000*	.4778973	.000	10.098782	12.629618
GROUP3	1	-16.4926667*	.4778973	.000	-17.758085	-15.227249
	2	5.1720000*	.4778973	.000	3.906582	6.437418
	4	16.5362000*	.4778973	.000	15.270782	17.801618
GROUP4	1	-33.0288667*	.4778973	.000	-34.294285	-31.763449
	2	-11.3642000*	.4778973	.000	-12.629618	-10.098782
	3	-16.5362000*	.4778973	.000	-17.801618	-15.270782

\*. The mean difference is significant at the 0.05 level.

FIG-1: Bar diagram showing Ra values in nm



#### Interpretation of the results

The unpolished group showed the highest mean surface roughness (Ra) value of 40.262000nm. The group 2 and 3 samples had the Ra values of 18.597333nm and 23.769333nm respectively. The Ra value for group 4 was 7.233133nm, which was the lowest among the others. The Ra values arranged in descending order is as follows; GROUP 1 > GROUP 3 > GROUP 2 > GROUP 4. Within group analysis of the Ra values was done using one- way ANOVA with Tukey's post hoc tests. These tests demonstrated a highly significant difference ( $p < .001$ ) between the mean surface roughness of all the 4 groups.

#### IV. Discussion

Proper finishing and polishing have been related to less plaque retention, consequently decreased secondary caries rate and marginal discoloration, thus enhancing the longevity and esthetics of the restoration. Surface roughness can be expressed as a function of the microrelief of the surface created during the finishing and polishing procedure<sup>5</sup>. During this process abrasion of resin matrix and filler particles can be accompanied: (i) by the softening of resin matrix due to the production of highly localized heat<sup>6</sup>; (ii) by the creation of residual defects and surface flaws caused by dislodgement or debonding of the glass fillers<sup>7,8</sup> and (iii) by scratch lines left by abrasives of greater size<sup>8</sup>. The microrelief of the surface especially voids, cracks and pits is of critical clinical relevance as it has been reported to create protected sites for bacteria<sup>9</sup>.

Polishing of dental composite is complicated by the heterogeneous nature with both hard filler particles and soft resin matrix<sup>10,11</sup>. Resin removal rather than glass filler abrasion during the polishing procedure contributes to the exposure of filler particles and increases the surface roughness<sup>5</sup>. In order to effectively polish a resin composite, an abrasive should remove the resin matrix as well as cut the relatively harder filler particles simultaneously. It has been suggested that the filler particle size, shape, hardness and load have the potential to influence the surface characteristics of a resin composite<sup>12,13</sup>.

In this study the nanocomposite material selected was Filtek Supreme Z250 XT which has a homogenous filler structure and is close to that of microfilled composite. The filler structure includes surface-modified zirconia/ silica with a mean particle size of approximately 3 $\mu$ m/less; non-agglomerated/ non-aggregated 20nm surface- modified silica particles and the filler loading is 82% (by wt.) or 68% (by

vol.). Previous studies<sup>14,15,16</sup> have shown that Filtek Supreme XT has produced smoother surface among all the 3 subclasses of nanocomposites. This result could be related to the specific composition of Filtek Supreme, which contains only nanofillers, which is in the same size range as the microfillers. The nanofillers are discretely dispersed or organized in clusters. These purely inorganic clusters are formed by individual primary nanoparticles bonded between them by weak intermolecular forces. Hence, these nanoparticles may break away from the clusters during wear or polishing.<sup>17,18,19</sup>

Many studies<sup>20,21,22</sup> reported that aluminium oxide discs gave smoother finish than diamond and silicon carbide polishing systems. This may be due to the size and hardness of the aluminium oxide particles incorporated in the polishing system to cut the filler particles and the resin matrix simultaneously.<sup>23,24</sup>

For analyzing the surface topography after polishing, AFM was used as it has got a higher resolution (in the level of nanometers) and capability to distinguish surface roughness than profilometer and SEM.<sup>25,26,27,28</sup> AFM images represents the surface morphology of the specimens caused by the exposed fillers. The high-resolution capacity of AFM permits accurate views of the surface topography, with 3D imaging of individual glass particles. The AFM calculated roughness comes as a complementary and local result to characterize the surfaces. AFM gives a higher lateral resolution (<30 nm) compared to optical profilometry (2µm) and a smaller surface size for investigation (10µm×10µm for AFM and 1000µm×1000µm for profilometry).

Chemical mechanical planarization (CMP) introduced by Monsanto in 1965 is used to produce mirror-like surfaces with no measurable subsurface damage.<sup>29</sup> CMP has been traditionally used in the field of engineering for procedures like semiconductor polishing, optical lithography, producing reflecting surfaces for mirrors, lenses and the planarization of computer chips. Colloidal silica with different particle sizes are predominantly used in the different CMP slurries. Various modifications have been done in the traditional colloidal silica slurry for improvements in CMP, like the reduction in particle size to produce nanosilica abrasive.

Rajiv et. al (2002)<sup>2</sup> stated that the nanosilica particle abrasive slurry have the smoothest finishing and polishing in chemical mechanical planarization. The nanosilica abrasives with average diameter of 80-90 nm were used to prepare polishing slurry for silicon wafers. Gaikwad et. al (2008)<sup>3</sup> reported that the silica nanoparticle with a diameter of 64 nm produced smoother surface on the tooth, which decreased the caries rate and Streptococcus mutans adherence. The colloidal nano-abrasive particles not only provides high polishing rate, but also achieves a very smooth surface.

In this study porous nanosilica is used which according to recent studies<sup>30,31,32</sup> are said to exhibit better surface planarization and fewer scratches than traditional solid nanosilica during the polishing. This porous nanosilica has a typical hexagonal mesoporous structure with a p6mm pore arrangement belonging to the SBA-15 family of porous structures.

The results of this study showed that the porous nanosilica produced the smoothest surface among the 4 groups. According to Rajiv et. al (2002)<sup>2</sup> and Gaikwad et. al (2008)<sup>3</sup>, when the particle size of the abrasive slurry was decreased (to the level of nanometers), the material removal from the particle may also be reduced due to lower stresses (in nanoscales). The degree of surface scratching may be decreased due to the reduced indentation as the abrasive particle size was smaller.

The results also showed that the Group 2 (Sof-lex) produced smoother surface than the Group 3 (Super-Snap) with statistical significance (p<0.05) which is in accordance with the results of previous studies conducted.<sup>19</sup> Increased smoothness of Sof-lex polished surface may be due to the fact that the abrasive particle size in Super-Snap (ultrafine disc has particle size of 8µm) is larger than that of Sof-lex (ultrafine is 7µm).

## V. Conclusion

Within the limitations of this in vitro study, the following conclusions were drawn: Composite restoration should be polished to produce a smooth surface. The smoothest surface was produced by porous nanosilica abrasive slurry than the commercially available polishing systems- Sof-lex and Super Snap.

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