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Research Article

EVALUATION OF DYNAMIC HARDNESS AND ELASTIC MODULUS OF FOUR RESIN COMPOSITES BY DYNAMIC MICRO-INDENTATION METHOD" -AN INVITRO STUDY

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A R T I C L E I N F O

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A B S T R A C T

Introduction: Restorative dentistry has became more exciting with the introduction of tooth coloured restorative materials such as composites and ceramics. Many advances in these materials have been introduced into the market in the recent past, making it difficult for a clinician to choose any particular type. The knowledge regarding mechanical properties like dynamic hardness, elastic modulus, wear resistance etc of a material is essential for a clinician to choose. Micro indentation hardness testing is a convenient way of investigating the mechanical properties of a small volume of material.
Aim: This study aims to compare dynamic hardness and elastic modulus of four commercially available resin composites.
Materials & Method: Four commercially available resin composites Filtek Z350xt, Swisstec, Charisma Smile, Tetric N-Ceram Were taken. Twenty disc shaped specimens were prepared from four resin composites and divided into four groups of five specimens each. The specimens were tested for mechanical behaviour using dynamic micro indentation hardness tester. **Results:** The results were analysed using SPSS version 23, descriptive statistics, ANOVA test with post hoc scheffe test done for intergroup comparison of the hardness and elastic modulus.
Conclusion: The dynamic micro-indentation method is a useful technique for determining the mechanical behavior of dental resin composites as brittle material. Within the limitations of this in vitro study, maximum dynamic hardness is seen in Charisma smile and the least dynamic hardness is seen in Filtek Z350XT. And maximum elastic modulus is seen in Charisma smile and the least dynamic hardness is seen in Swisstec.

INTRODUCTION

Restorative dentistry has become more exciting because of the introduction of tooth coloured restorative materials such as the composites and the ceramics. Many advances in these materials have been introduced into the market in the recent past, making it difficult for a clinician to choose any particular type. The knowledge regarding mechanical properties like dynamic hardness, elastic modulus, wear resistance etc of a material is essential for a clinician to choose.

Dental resin composites are widely used as restorative materials because they are esthetically pleasing and relatively easy to handle in restorative treatment.[1,2] Accordingly, in clinical situations, resin composites have been reported to adapt well to the cavity wall due to their low viscosity.[3]

For clinicians, the use of a composite with a specific consistency in restorative treatment is attractive because the viscosity affects the application and manipulation of the material. The strength of composites in the oral environment as well as the handling characteristics of composites in restorative procedures is important.[4] Various strengths of composites have been evaluated by different mechanical testing techniques.[5]

Ilie and Hickel compared the mechanical properties, such as flexural strength, compressive strength, and tensile strength, of 72 commercially available restorative materials. They reported that a good correlation was only found between the flexural strength and flexural modulus, which was also correlated with the filler volume, whereas compressive strength and tensile strength were less sensitive. Strength is the maximum stress required to fracture a structure or material. Generally, the high compressive strength of resin composites does not imply high tensile strength, reflecting somewhat brittle nature of resin composites. Thus, the measurement of the tensile strength of brittle materials, such as resin composites, is extremely difficult.[6]

To determine the mechanical properties, indentation hardness tests are the most commonly used non-destructive testing procedures in the metal industry and in research because they

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provide an easy, inexpensive, and reliable method to evaluate the basic properties of developed or new materials. The indentation hardness of materials is measured in several ways by forcing an indenter with a specific geometry, such as a ball, cone, or pyramid, into the specimen's surface.[7,8]

Micro-indentation hardness testing is a convenient way of investigating the mechanical properties of small volume of material. Micro-indentation method has significant advantages over conventional hardness testing. In this depth-sensing indentation hardness test, a diamond indenter tip is pressed into the specimen until a given maximum load or depth and then removed. The load on and the displacement of the indenter are simultaneously recorded. This method can provide well-defined mechanical parameters of materials, such as the dynamic hardness and the elastic modulus.[9]

In this study, we compared the dynamic hardness and elastic modulus of four different resin composites using dynamic micro-indentation method.

AIM

This study aims to compare dynamic hardness and elastic modulus of four commercially available resin composites; FILTEK Z350XT, SWISSTEC, CHARISMA SMILE, TETRIC N-CERAM.

MATERIALS AND METHOD

There was preparation of four commercially available resin composites. i.e FILTEK Z350XT, SWISSTEC, CHARISMA SMILE, TETRIC N-CERAM. All materials were of shade A1 (Fig 1a,b,c,d).

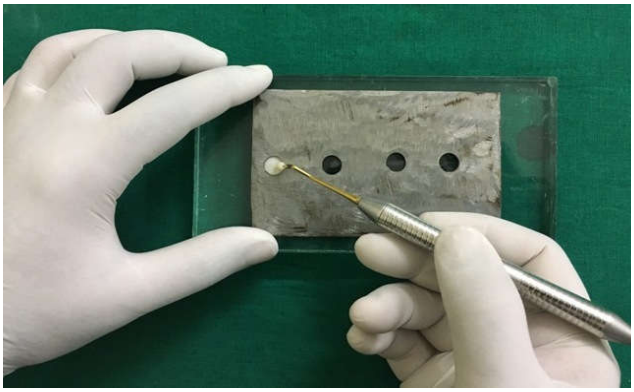
Fig 1(a): FILTEK Z350XT

Fig 1(b): SWISSTEC

Fig 1(c): CHARISMA SMILE

Fig 1(d): TETRIC N-CERAM

Standardized stainless steel moulds are fabricated to acquire the composite blocks (8.5mm diameter & 3mm depth). The mould is placed on transparent glass slab and mylar strip. The mould space is filled with resin composite with incremental technique till the mould space is filled completely (Fig 2a). Another mylar strip and glass slab is used to pack the composite and cured for 40 seconds using light cure unit (QHL75 Curing Light; Dentsply sirona, USA) to ensure complete polymerization of the composite (Fig 2b,c).[10]



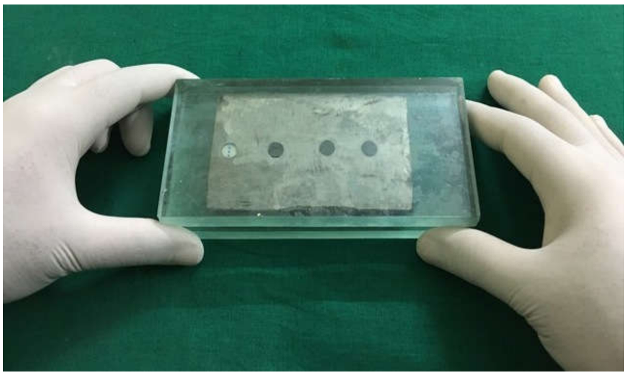


Fig 2(a) Mould space is filled with composite **Fig 2(b);** Mylar strip and glass slab is used to pack the composite

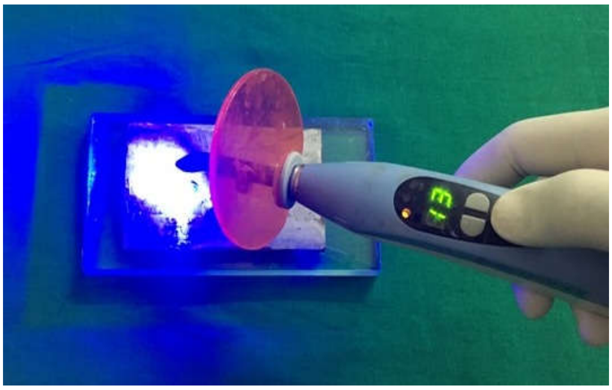


Fig 2(c) Composite is cured for 40 seconds

After polymerization, the composite block was carefully removed from the mould. Both surfaces of the composite were polished using #800 and #1000 silicon carbide paper under running water to remove residual monomers. To fabricate the test specimens for the dynamic micro-indentation test, twenty disc shaped specimens were prepared from four resin composites and divided into four groups of five specimens each (Fig 3). Finally, the four resin composites were stored in distilled water for 24 h at 37°C.

Fig 3 Disc shaped specimens of four resin composites

Dynamic micro-indentation test

The investigation of the mechanical behavior of the resin composites was performed using a dynamic ultra-micro-hardness tester (DUH-211; Shimadzu Co., Kyoto, Japan) fitted with a Berkovich indenter tip (Figure 4a). The specimens were fixed onto an attached holder. Dynamic micro-indentation primarily involves applying a controlled load (*P*) through a diamond tip that is in contact with a smooth surface. The penetration depth (*h*) of indentation is continuously recorded as a function of load. Figure 4(b) shows a schematic illustration of the typical micro-indentation load- penetration depth curve obtained from dynamic micro-indentation tests.

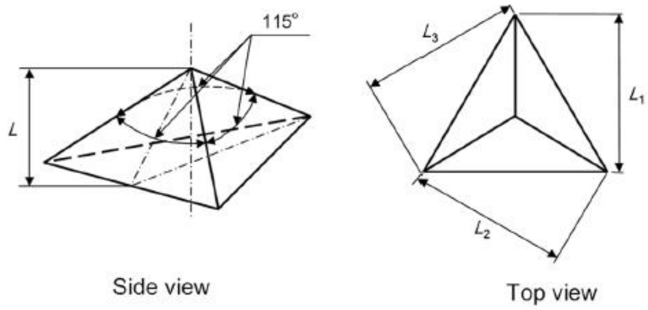


Fig 4(a)

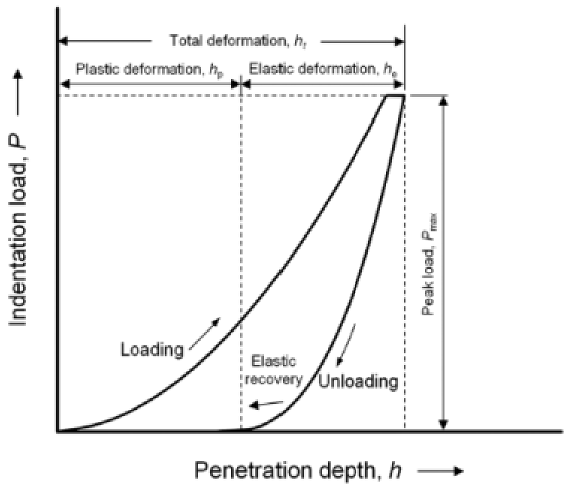


Fig 4(b)

Fig 4 Dynamic ultra-micro-hardness testing system used in this study. (a) Schematic image of the geometry of the Berkovich indenter tip. Tip height (*L*) is defined as $L = (1/3)(L1+L2+L3)$. The photograph is the indented surface of the sample after testing. (b) Schematic illustration of the micro-indentation load versus penetration depth curve obtained from the testing.

During indenter loading and unloading, the specimen is subjected to both plastic deformation (*h*p) and elastic deformation (*h*e). The total deformation (*h*t) is the sum of *hp* and *he* in the micro-indentation load-penetration depth curve.

Moreover, the dynamic hardness and elastic modulus can be obtained from the indentation load and penetration depth data.[11]

The dynamic hardness (*DH*) of the sample was calculated from the following equation

$$DH = \frac{\alpha P}{h^2}$$

where α is a geometrical constant of the Berkovich indenter (3.8584), *P* is the applied load during the indentation test, and *h* is the penetration depth of indentation.

The elastic modulus (E) of the sample was calculated from the following equation

$$\frac{1}{\mathrm{E_r}} = \frac{(1-\mathrm{V}^2)}{E} + \frac{(1-\mathrm{V_i}^2)}{E_i}$$

where Er is the reduced elastic modulus from the indenter, V is Poisson's ratio for the sample, Vi is Poisson's ratio for the Berkovich indenter (0.07), and Ei is the modulus of the Berkovich indenter (1140 GPa).

In this study, the dynamic micro-indentation test was carried out with peak loads (Pmax). The load rate was kept constant at 15 mN/s, and the hold time at the maximum load was set to 5 sec. The dynamic micro-indentation results, such as dynamic hardness and elastic modulus, were obtained as the average values of all specimens (Fig 5).

Fig 5 Dynamic micro-indentation tester

Statistical analysis

The results obtained from the dynamic micro-indentation tests were analysed using SPSS version 23, descriptive statistics, ANOVA test with post hoc scheffe test done for intergroup comparison of the hardness and elastic modulus.

RESULTS

Table 1 Intergroup comparison of Micro hardness (one way ANOVA)

Group	N	Minimum	Maximum	Mean	Std. Deviation	F value	P value
Filtek Z350XT	5	49.11	128.92	83.07	25.99	119.36	<0.001**
Swisstec	5	199.21	290.28	233.11	31.08		
Charisma smile	5	593.74	967.05	765.32	125.38		
Tetric N- Ceram	5	84.99	192.34	133.63	40.45		

**-Highly significant (p<0.001), *-Significant (p<0.05), NS- Not significant (p>0.05)

There is a statistically significant difference present in the hardness among various groups, with maximum hardness with Charisma smile and the least hardness with Filtek Z350XT. (ANOVA signifies overall comparison, to know individual pairwise comparisons Post Hoc Scheffe test should be done).

Table 2 Post hoc scheffe test for individual pair wise comparison

Comparison Between		Mean Difference	P Value
Filtek Z350 XT	SwissTec	-150.04	0.015*
Filtek Z350 XT	Charisma Smile	-682.25	<0.001**
Filtek Z350 XT	Tetric N- Ceram	-50.56	0.593 NS
SwissTec	Charisma Smile	-532.21	<0.001**
SwissTec	Tetric N- Ceram	99.48	<0.001**
Charisma Smile	Tetric N- Ceram	631.69	<0.001**

**-Highly significant (p<0.001), *-Significant (p<0.05), NS- Not significant (p>0.05)

There is no statistically significant difference in the hardness between Filtek and Tetric N-Ceram, all the other comparison showed statistically significant difference.

The order of hardness in the compared group is

Charisma Smile > SwissTec > Tetric N-Ceram > Filtek.

Table 3 Intergroup comparison of Elastic modulus (one Way ANOVA)

Group	N	Minimum	Maximum	Mean	Std.Deviation	F value	P value
Filtek Z350XT	5	5.71	15.55	10.09	3.67	6.613	<0.004*
Swisstec	5	6.19	6.85	6.52	0.23		
Charisma smile	5	10.05	12.40	11.35	0.85		
Tetric N-Ceram	5	6.88	8.76	7.65	0.72		

**-Highly significant (p<0.001), *-Significant (p<0.05), NS- Not significant (p>0.05)

There is statistically significant difference present in the elastic modulus among various groups. With maximum elastic modulus with Charisma smile and the least elastic modulus with Swisstec. (Anova signifies overall comparison, To know individual pairwise comparisons Post Hoc Scheffe test should be done).

Table 4 Post hoc scheffe test for individual pair wise comparison of elastic modulus

Comparison Between		Mean Difference	P Value
Filtek Z350 XT	SwissTec	3.57	0.043*
Filtek Z350 XT	Charisma Smile	-1.26	0.730 NS
Filtek Z350 XT	Tetric N- Ceram	2.44	0.225NS
SwissTec	Charisma Smile	-4.83	0.005*
SwissTec	Tetric N- Ceram	-1.13	0.789NS
Charisma Smile	Tetric N- Ceram	3.7	0.035*

**-Highly significant (p<0.001), *-Significant (p<0.05), NS- Not significant (p>0.05)

Order of elastic modulus:
Charisma smile > Filtek > Tetric N-Ceram > Swisstec

DISCUSSION

It is important for clinicians to have reliable information on the mechanical properties of commercially available resin composites, as well as the handling characteristics in restorative procedures. Micro-indentation is a depth-sensing technique that can accurately characterize the mechanical properties of almost all types of solid materials at a small scale.

In particular, this method can continuously record the penetration depth of the specimen during dynamic loading and unloading at the indentation tip while providing information on the degree of plastic and elastic deformation, and the hardness and elastic modulus of the materials.

Therefore, knowledge of the mechanical properties of such resin composites provided by micro-indentation is very helpful for clinicians in relation to restorative treatment. In this study, the micro-indentation method was used to investigate the mechanical properties, such as dynamic hardness and elastic modulus, of four commercially available resin composites.

Dental composites are normally composed of a dispersion of silica filler within a resin matrix. To enhance the chemical bonding between the silica glass and dimethacrylate, the silica

glass is treated with a silane coupling agent, which has a methacryloyl group at its terminal end.
Sabbagh *et al*[13] reported that some manufacturers assess the weight percentage of fillers before the silanization of the fillers, whereas others include the percentage of silane coating in their calculations. As a result, it is thought that the differences between experimental filler content and those given by manufacturers can be attributed to filler silanization.[14]

By considering the recent advances in nanotechnology, nanofill composites contain silica and zirconia nanoparticles, which are partially calcined inorder to produce micron-sized porous clusters that are infiltrated with silane prior to incorporation into a resin matrix.[9,10] Therefore, it is expected that if there is more silane coupling agent then the surface area of the filler particles increases with decreasing particle size of the filler.[15]

Although the filler content generally affects the mechanical properties of composites, there was no correlation between either dynamic hardness or elastic modulus and the inorganic-filler content of the resin composites.

In this study there is statistically significant difference present in the dynamic hardness among various groups, with maximum hardness seen in Charisma smile and the least hardness in Filtek. And there is statistically significant difference present in the elastic modulus between various groups, with maximum elastic modulus seen in Charisma smile and the least elastic modulus in Swisstec.

Finally, it is well known that the mechanical properties of composites depend on stress transmission at the interphase between the silica filler and the resin matrix in the composites. Thus, optimization of the mechanical properties of composites is based on knowledge of the relationship between the microstructure and the macroscopic response. Finite element analysis (FEA) has the potential to predict the mechanical behavior of dental composites, which can be microscopically divided into the filler, the matrix, and the interphase.[16]

To analyze the mechanical characterization of composites by FEA, the material properties of the filler reinforcement, resin matrix, and interphase components are required. In particular, the elastic modulus is a basic input parameter for all of the components in computational FEA.

Ho and Marcolongo evaluated the interfacial mechanics of bioactive composites composed of polymethylmethacrylate and hydroxyapatite for mandibular bone substitutes using the nano-indentation technique. They concluded that nano-indentation is a powerful technique for quantifying interfacial interactions in composites and is capable of showing more specific local differences in the interfacial mechanics than the quasi-static bulk properties, such as flexural bending.[17]

In this study, the main advantage of the dynamic micro-indentation test compared with other mechanical test procedures for resin composites is the relative simplicity of the experimental setup. The material properties of the components, such as the filler reinforcement, the resin matrix, and the interphase, will be further investigated by the dynamic micro-indentation method.

CONCLUSION

This study compares the mechanical behavior of four commercially available resin composites by the dynamic micro-indentation method. The following conclusions were drawn:

- Within the limitations of this *in vitro* study, it may be concluded that maximum dynamic hardness is seen in Charisma smile and the least dynamic hardness is seen in Filtek Z350XT.
- And maximum elastic modulus is seen in Charisma smile and the least dynamic hardness is seen in Swisstec

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