INTRODUCTION

The restoration of endodontically treated or severely broken down teeth, which in most cases are mechanically compromised, is required to compensate for the considerable bulk loss of hard tissues. There are many studies that have focused on the restoration of teeth after endodontic procedures1,2). A core build-up is a restoration that facilitates a subsequent extracoronal restoration. Traditionally, metal cores were used2), but because of the difference in elastic moduli between metal and dentin, stress concentrations leading to root fractures resulted3). To overcome this problem, composite resin materials that match the dentin modulus were developed4). According to a very recent nationwide survey in Germany, adhesive composite core build-ups were the predominant treatment approach to restore endodontically treated teeth (75%) 5). However, defects and flaws in these materials often lead to catastrophic failures6). In the constant pursuit of improved mechanical properties, manufacturers are continually releasing materials with new and advanced formulations. The performances of these materials under oral conditions are related to their physicomechanical properties. Undoubtedly, the ability of a core build-up restoration to tolerate fractures is related to its composition and filler volume. It has been demonstrated that increased filler loading at a threshold of approximately 55–57% by volume resulted in higher fracture toughness7-9). Because of the great variety and complex nature of forces in the oral cavity, many different methods for the mechanical characterization of restorative materials have traditionally been employed. These include three-point bending (ISO 4049), tensile fracture strength testing10), and indentation and nanoindentation hardness methods11,12). Fracture toughness ($K_{IC}$) is one of the most important measures of a material’s properties and is a direct indication of the amount of stress that a material is capable of bearing prior to failure. There are many methods for assessing $K_{IC}$; single-notch and chevron-notched rods are most commonly employed13). Recently, a fracture toughness method using notchless triangular prisms (NTPs) as specimens was proposed to avoid the inconvenience and shortcomings associated with placing a notch14).

The aim of this study was to evaluate a newly developed experimental direct core build-up material mechanically and micromechanically with different amounts of filler loading (+2.5 and +5 wt%). The null hypothesis tested was that there is no difference in mechanical or micromechanical performance between materials with variable filler loading contents.

MATERIALS AND METHODS

This study employed an experimental core build-up material based on the commercially available Clearfil DC Core Automix One (74 wt% filler load, Kuraray Noritake Dental, Tokyo, Japan) with two different amounts of filler content presented as weight percentages (+2.5 and +5 wt%). The null hypothesis tested was that there is no difference in mechanical or micromechanical performance between materials with variable filler loading contents.
Table 1  Chemical compositions

| Product name [code] and manufacturer                      | Materials [lot no.] | Components                                           |
|-----------------------------------------------------------|---------------------|------------------------------------------------------|
| Clearfil DC Core Automix One [DC] (filler load 74 wt%)    | 6Q0202              | silanated barium glass filler, silanated silica,   |
| Kuraray Noritake Dental, Tokyo, Japan                     |                     | Bis-GMA, TEGDMA, CQ, chemical catalyst, accelerators|
| Clearfil DC Core Automix One [DC + 2.5 wt% filler]       | 160218              | silanated barium glass filler, silanated silica,   |
| Kuraray Noritake Dental                                    |                     | Bis-GMA, TEGDMA, CQ, chemical catalyst, accelerators|
| Clearfil DC Core Automix One [DC + 5 wt% filler]         | 160219              | silanated barium glass filler, silanated silica,   |
| Kuraray Noritake Dental                                    |                     | Bis-GMA, TEGDMA, CQ, chemical catalyst, accelerators|

Bis-GMA: bisphenol-A-diglycidyl methacrylate; TEGDMA: triethyleneglycol dimethacrylate; CQ: camphorquinone

universal testing machine (Autograph AG-IS 20 KN, Shimadzu, Kyoto, Japan) conforming to the ISO 4049 standard. Twenty beam-shaped specimens of each material were obtained using a custom-made metallic mold (2x2x25 mm) pressed between transparent plastic strips and glass to extrude excess material. After light polymerization for 4x20 s from the top and bottom sides with an LED device (wavelength, 380–430 nm; light output intensity, 1,000+400 mW/cm²; PenCure 2000; J. Morita Mfg., Kyoto, Japan) and storage in distilled water at 37°C for 24 h, the specimens were subjected to complete failure in a three-point bending test at a crosshead speed of 0.5 mm/min. The flexural fracture strength was calculated using the following equation:

\[ S_{\text{max}} = \frac{3Pl}{bd^2} \]

where \( P \) is the added load, \( l \) is the outer span length, \( b \) is the specimen width, and \( d \) is the specimen thickness.

Fracture toughness (Kic)

Twenty NTP specimens were prepared for each material group by using a custom-made metallic mold (6x6x6x12 mm) under the same pressing, polymerizing, and storage protocol as for flexural fracture testing. The specimens were then secured in custom-designed grips and subjected to complete failure in tensile load mode (EZ-Test, Shimadzu) at a crosshead speed of 0.1 mm/min. Each sample was scored at the location of the tensile forces to create an approximately 0.1-mm-deep crack initiation point. The relationships that were used to calculate the \( K_{\text{ic}} \) value were proposed by Barker15) and adopted by ASTM standard E1304. The equation is as follows:

\[ K_{\text{ic}} = \frac{P_{\text{max}}}{DNW} Y_{\text{min}} \]

where: \( P_{\text{max}} \)=the maximum load at fracture, \( D \)=the specimen diameter (12 mm), \( W \)=the specimen length (10.5 mm), and \( Y_{\text{min}} \)=the minimum of the dimensionless stress intensity factor coefficient (=28) as reported by Bubsey et al.10) and proposed by Ruse et al.14). The assembly of the NTP specimen and specimen holder is shown in Fig. 1.

Indentation hardness (HIT) and the reduced elastic modulus (EIT)

Three specimens with approximate sizes of 4x4x2 mm were prepared (one for each material). The large surfaces were then fine-polished using a polishing device (EcoMet 3000, Buehler, Lake Bluff, IL, USA) using 800, 1000, 1200, 1500, and 2000-grit SiC papers and paper polishing pads under alumina polishing paste slurries of particles with decreasing sizes (1, 0.3, and 0.05 μm) to create a smooth surface suitable for the nanoindentation study. The specimens were then inspected under a stereoscopic zoom microscope (SM-Z 200, Nikon, Tokyo, Japan) prior to testing to check the integrity and quality of the polished specimens. The nanohardness and indentation elastic moduli of the resin composite were estimated by nanoindentation equipment (Nanoindenter G200, Keysight Technologies, Santa Rosa, CA, USA). The method of indentation used in this study is a load controlled method and the loading program is cycles loading with number of cycles equal to five. The indenter tip approaches the surface and when the indenter senses the surface, the cyclic loading/unloading begins. For each cycle, the indenter penetrates the surface for 10 s until reaching the maximum load for the current cycle followed by 10 s hold time and unloading of 90% of the maximum applied load reached during the current loading cycle. After the last cycle, the final load at unloading is held constant for 75 s to account for the thermal drift. The indenter is then withdrawn completely and ready to move into position for the next indentation test.

The maximum loads in all tests were 500 mN (nine indentations per sample; the maximum force achieved within five loading-unloading cycles). A distance of 100 μm was allowed between indentations to avoid any influence of residual stresses from adjacent indentations. The indentation modulus \( E_{\text{IT}} \) and hardness \( H_{\text{IT}} \) were calculated using the unloading part of the measured
value from the indentation test load-displacement curve as shown in Fig. 2. The equations to derive these mechanical characteristics are\textsuperscript{17}:

\[ H_{IT} = \frac{P_{\text{max}}}{A(h_c)} , \]  \hspace{1cm} \text{(1)}

\[ E_{IT} = (1 - \nu_s^2) \left[ \frac{2\beta}{S} \sqrt{\frac{A(h_c)}{\pi}} - \frac{1 - \nu_i^2}{E_i} \right]^{-1} \]  \hspace{1cm} \text{(2)}

For a Berkovich indenter tip, the coefficient $\beta=1.034$, $P_{\text{max}}$ is the maximal applied force, $\nu_s$ is the Poisson ratio for the specimen (in our case, $\nu_s=0.25$), and $\nu_i$ and $E_i$ are the Poisson ratio and elastic modulus of the diamond tip, respectively. The projected area of contact between the indenter and specimen as a function of the contact depth $h_c$ is introduced by the following approximation\textsuperscript{17}:

\[ A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16} \]  \hspace{1cm} \text{(3)}

The coefficients in equation (3) are determined by a calibration procedure that consists of a nanoindentation experiment on a standardized quartz sample (fused silica) with a known elastic modulus and hardness independent of the indentation depth.

**Weibull analysis**

The Weibull statistical analysis method was used to estimate the characteristic $K_{IC}$, characteristic fracture strength, and reliability of each material. In the Weibull statistical analysis, the probability of fracture $P_f$ is related to the fracture stress $\sigma$ by the following exponential relationship\textsuperscript{18}:

\[ A(h_c) = C_0 h_c^2 + C_1 h_c + C_2 h_c^{1/2} + C_3 h_c^{1/4} + C_4 h_c^{1/8} + C_5 h_c^{1/16} \]  \hspace{1cm} \text{(3)}

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\[ P_f = 1 - e^{\left[ -\frac{(\sigma - \sigma_u)}{\sigma_\theta} \right]^m} \]

This is known as the Weibull three-parameter strength distribution. The threshold stress parameter, \( \sigma_u \), represents the minimum stress below which a test specimen will not break. The scale parameter or characteristic strength \( \sigma_\theta \) is dependent on the stress configuration and test specimen size. The distribution shape parameter \( m \) is the Weibull modulus. In order to analyze the results with Weibull statistics, 20 specimens were prepared for each group19). The Weibull modulus \( (m) \), represented by the slope of the curves, indicates the reliability of each material (the steeper the curve, the more reliable the material). Characteristic Weibull \( K_{IC} \) and Weibull fracture strength values were obtained from the plot at the 63.2nd percentile. An ANOVA and Tukey’s test \((\alpha<0.05)\) were also performed to compare the results.

**Scanning electron microscopy (SEM) observations of the fractured surfaces**

Selected fractured specimens from the flexural fracture testing and NTP tensile testing were sputter-coated for SEM (JEOL JSM 6390 LV, Tokyo, Japan) observation of the fractured surfaces. All fractured surfaces were observed under SEM with magnifications ranging from \( \times50 \) to \( \times5,000 \).

### RESULTS

The results are summarized in Tables 2, 3, and 4 and in the Weibull plot in Fig. 3. The different superscript letters indicate statistically significant difference between groups \((\alpha\leq0.05)\). Generally, with an increase in filler content, the fracture strength increased significantly from 91.72±11.8 to 114.10±9.1 MPa for DC+2.5 wt% and to 116.64±14.7 MPa for DC+5 wt%. The same tendency was observed for the elastic moduli and indentation hardness. The elastic moduli increased from 12.63±0.92 to 15.36±1.2 GPa for DC+2.5 wt% and to 15.31 GPa for DC+5 wt%, while the indentation hardness increased from 0.45±0.03 to 0.55±0.04 and 0.57±0.11 GPa for DC+2.5 wt% and DC+5 wt%, respectively. The probabilities of fracture for the tested groups of specimens are presented in Fig. 3 below. The Weibull moduli \( (m) \), defined by the slopes of the curves, showed that the DC+2.5 wt% specimens were the most reliable in both the fracture strength and fracture toughness tests.

Figure 4 shows typical examples of fractured specimen surfaces from both fracture strength and NTP fracture toughness testing. Note the air bubbles inclusions with the increase of filler amount (arrowed).

The indentation imprints, indentation moduli, and hardnesses for five different penetration loads are presented in Fig. 5 below.

The materials were tested in cyclic mode under five

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**Table 2** Fracture strength and Weibull statistical analyses of core buildup materials

| Material       | Fracture strength (MPa) Mean (SD) | Weibull modulus \( (m) \) | Characteristic Weibull fracture strength (in MPa) |
|----------------|-----------------------------------|----------------------------|-----------------------------------------------|
| DC             | 91.72 (11.8)\(^a\)               | 9.28                       | 4.68                                          |
| DC+2.5 wt%     | 114.10 (9.1)\(^b\)               | 15.03                      | 4.84                                          |
| DC+5 wt%       | 116.64 (14.7)\(^b\)              | 9.47                       | 4.92                                          |

**Table 3** Fracture toughness \( (K_{IC}) \) and Weibull statistical analyses of core materials

| Material       | \( K_{IC} \) (in MPa•m\(^{1/2}\)) Mean (SD) | Weibull modulus \( (m) \) | Characteristic Weibull \( K_{IC} \) (in MPa•m\(^{1/2}\)) |
|----------------|-----------------------------------------------|----------------------------|-------------------------------------------------------------|
| DC             | 1.82 (0.60)\(^a\)                            | 3.00                       | 1.05                                                        |
| DC+2.5 wt%     | 2.28 (0.53)\(^b\)                            | 5.18                       | 1.10                                                        |
| DC+5 wt%       | 2.68 (0.88)\(^b\)                            | 3.66                       | 1.37                                                        |

**Table 4** Indentation hardness \( (H_{IT}) \) and reduced elastic moduli \( (E_{IT}) \) of core materials

| Material       | Indentation hardness \( H_{IT} \) (in GPa) Mean (SD) | Reduced elastic modulus \( E_{IT} \) (in GPa) Mean (SD) |
|----------------|-------------------------------------------------------|---------------------------------------------------------|
| DC             | 0.45 (0.03)\(^a\)                                     | 12.63 (0.92)\(^a\)                                      |
| DC+2.5 wt%     | 0.55 (0.04)\(^b\)                                     | 15.36 (1.20)\(^b\)                                      |
| DC+5 wt%       | 0.57 (0.11)\(^b\)                                     | 15.31 (2.01)\(^b\)                                      |
different loads to investigate the presence of eventual inhomogeneity with depth.

**DISCUSSION**

The three-point bending test is based on ISO specification 4049:2000 for polymer-based restorative materials and is commonly employed in dental materials research\(^{20-22}\). This flexural bending test is usually recommended because the fabrication of specimens and load application are quite simple.

Fracture is one of the main reasons for failures of dental composites in clinical practice\(^{23}\). This usually occurs through pre-existing cracks and irregularities under tensile stress. These irregularities may be due to microstructural imperfections or bubble inclusions during mixing or insertion of the material\(^{24}\).

In this study, the fracture strength was associated with the filler content of the core build-up material. Generally, an increase in fracture strength was observed
Fig. 5  Indentation imprints a); moduli, and hardnesses under five different penetration loads b).

with an increase in filler content in wt% (ANOVA and Tukey’s test; \(p<0.05\)). The observed increases as compared with the control group were 24 and 28% for DC+2.5 and DC+5 wt%, respectively. This is in good agreement with the findings of Kim et al., who associated pre-polymerized filler particles with low filler content and poor mechanical properties\(^8\). Furthermore, the presence of TEGDMA in the composite matrix composition has been linked to a significant decrease in the flexural strength of the material; surprisingly, it has also been linked to an increase in the modulus of elasticity\(^25\).

The \(K_{IC}\) values were determined in this study by using a relatively new and effective NTP method, which includes a simple and reproducible procedure for specimen preparation. The tendency here was similar to that observed for fracture strength. A 5 wt% increase in filler led to a 47% tougher material as compared with the control (ANOVA and Tukey’s test; \(p<0.05\)).

The results from the nanoindentation testing were also affected by filler content. Figure 5 depicts the imprints and surfaces of three samples. The calculated indentation moduli and hardnesses under five different loads are also shown in Fig. 5. It can be concluded that the mechanical characteristics of the samples with increased filler contents were superior compared with those of the control. The samples with higher filler content had a more homogeneous distribution of the elastic modulus and hardness with depth. There was no significant difference in the mechanical properties of the samples with increased filler content but we can still speculate that the DC+5 wt% material with the higher filler content was stiffer than the DC+2.5 wt% material.

The Weibull statistical analysis was used to estimate the characteristic \(K_{IC}\), characteristic fracture strength, and reliability of each material. It is often noted that dental restorative composites, while popular in terms of esthetics and biocompatibility, are susceptible to brittle fracture\(^18\). For these materials, Weibull statistics are recommended. The Weibull modulus (\(m\)), given by the slopes of the curves (Fig. 3), indicates the reliability of each material (the steeper the curve, the more reliable the material). The addition of 2.5 wt% filler content led to the most reliable material.

SEM observation of the fractured surfaces from NTP testing revealed some bubble inclusions with increased filler content. This might be a possible explanation for the not-so-reliable nature of the DC+5 wt% material. On the other hand, the manipulative and handling abilities of the materials were not affected by the addition of filler particles.

Generally, the mechanical properties improved with an increase in filler content. There are some plausible mechanisms for this phenomenon. Some authors explain it with the crack bowing effect, resulting from pinning by the filler particles and increased energy at the crack tip\(^26,27\), while others suggest crack branching, thereby increasing the crack surface area and resultant energy\(^28\).

Although, the present study aims to focus at the micro-mechanical properties of core build-up materials, some effects of the filler contents regarding adhesion to the tooth substrate and polymerization are worth discussing. High inorganic contents were associated with low polymerization stress values, which is attributable
to the reduced shrinkage in highly filled composites. Our specimen preparation protocol employed light curing from top and bottom sides. This actually differs from the procedure of polymerization that is typically employed in a clinical situation, where it is being done from one side only. A recent study revealed that in Clearfil DC Core Automix One, the degree of conversion showed no statistical difference between self and dual cured modes.

CONCLUSION

Within the limitations of this study, it can be concluded that an increase in filler contents with 2.5 and 5 wt% of the control core build-up material (Clearfil DC Core Automix One) led to improved mechanical properties. Regarding reliability, the 2.5 wt% increase in filler yielded the most reliable material and could be the preferred choice for clinicians when strength and toughness are of primary importance. Further studies are also warranted to reveal the clinical longevity of these materials.

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