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Inorganic and Prepolymerized Filler Analysis of Four Resin Composites

DC Salazar • J Dennison • P Yaman

Clinical Relevance

Selection of a composite resin based on high filler content, may be misleading. The reported filler content may vary, depending on the ratio of prepolymerized resin to inorganic glass particles. A composite material should be labeled with two values: total filler and inorganic filler contents.

SUMMARY

This study determined the filler content by weight percentage of four resin composites and examined the morphology, size, and elemental distribution of the filler particles. Four commercially available light-cured resin composites were evaluated for filler content by weight using ashing in air and acetone dissolution techniques. Ten specimens were analyzed for each material and technique. Specimens for ashing were heated to 650°C for 30 minutes. For the acetone dilution, the uncured specimens were dissolved, centrifuged, and decanted. In addition, scanning electron microscopy evaluation and energy dispersive x-ray spectroscopy analysis were performed to determine morphologic characteristics and elemental distribution, respectively. Filler percentages by weight for Aelite LS, Filtek LS, IPS Empress Direct, and Kalore from ashed in air were 86.44%, 77.86%, 72.17%, and 70.62%, and from acetone dissolution percentages were 85.05%, 75.56%, 78.88%, and 77.73%, respectively. Aelite LS had significantly higher filler content for both techniques. Kalore had significantly lower filler content for the ashing technique (70.62%), and Filtek LS had significantly lower filler content for the acetone dissolution technique (75.55%). Manufacturer reported filler content for Aelite LS (88%) and Filtek LS (76%) approximated the study results for both techniques, while Kalore (82%) and IPS Empress Direct (79%) were only similar for acetone dissolution, indicating higher content of prepolymerized particles. Morphologic examination showed spherical shaped particles for Aelite LS and splintered and irregular shaped particles for all other materials. Aelite LS had the highest filler content for both techniques. Values for filler content by weight using the acetone dissolution were closer to manufacturer reported values.

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*Daisy C Salazar, DDS, MS, Department of Cariology, Restorative Sciences and Endodontics, University of Michigan, Ann Arbor, MI, USA

Joseph Dennison, DDS, MS, Department of Cariology, Restorative Sciences and Endodontics, University of Michigan, Ann Arbor, MI, USA

Peter Yaman, DDS. MS. Department of Cariology, Restorative Sciences and Endodontics, University of Michigan, Ann Arbor, MI, USA

*Corresponding author: 1011 N. University Ave, Ann Arbor, MI 48109; e-mail: daisysal@umich.edu

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INTRODUCTION

Since the first dental resin composites were developed, many efforts to improve their clinical performance have been undertaken. Researchers have suggested that filler content, size, and morphology of the filler particles within a composite resin formulation have the potential to influence the strength, elastic modulus, wear resistance, color matching, and polymerization shrinkage of a composite resin.[1-8] In addition, researchers have reported that increasing the filler particle size will effectively modify not only the pattern and rate of wear, but the restoration's polishability as well. It has also been stated that the greater the particle's size, the greater the potential for wear, which in turn affects mechanical properties of composites.[3,8-10] Thus, it would seem reasonable to expect more studies reporting correlations between mechanical properties and filler particle morphology and size. Perhaps the lack of some investigations is due to the difficulty in determining the exact size of the filler particles within a composite resin.[11] Furthermore, attempts to improve clinical performance and to decrease polymerization stress of methacrylate-based composites have focused on the development of new monomers, such as ring-opening silorane chemistry[12-14] and a new nanofiller technology.[7,15,16]

Only one study has reported measuring filler content by weight for current commercial composites using a technique that preserves prepolymerized particles.[17] Furthermore, there is no standard procedure for verifying a manufacturer's report of filler loading and size except by the least expensive method of ashing in air.[18] Ashing in air is a standard technique that has been used in numerous studies to determine filler content by weight.

Several methods have been suggested to determine the filler loading in resin-based composites: thermogravimetric analysis, gas pycnometry, or ashing in air. Ideally, filler loading should not vary with the different test methods. However, different results have been reported by varying the test method among resin-based composite materials. Factors like organic matrix and inorganic fillers as well as silane coating and prepolymerized particles have been reported to influence the filler content analyses of these materials.[8-10,19-21]

Scanning electron microscopy (SEM) often uses a dissolution technique with acetone or ethyl alcohol to remove the organic matrix from inorganic fillers.[3,8-10,22-25] According to some manufacturers, ashing in air can burn off some of the filler content of composites and thus give false results. To combat this problem, a separation of the matrix and filler using acetone needs to be explored. It is hypothesized that a solvent such as acetone will not break down prepolymerized filler, silane, agglomerates, or clusters from composite formulations. Acetone dilution has been used to remove the organic matrix for SEM analysis in many studies but has not been addressed as a possible technique to determine filler by weight. A solvent such as acetone is an alternative to determine filler by weight when including prepolymerized particles.[17]

The aim of this study was to determine the filler by weight percentage of four different resin composites using both ashing in air and acetone dissolution techniques. A secondary aim is to examine the morphology and composition of the filler particles in each material.

MATERIALS AND METHODS

Four commercially available resin composites (Table 1) were chosen based on their reported filler content by particle size and evaluated in this study for filler content by weight percentage. In addition, energy dispersive x-ray spectroscopy (EDS) analysis and SEM morphologic characterization were performed on the filler particles. Filler percentages obtained were compared against manufacturer's data.

Ten specimens were analyzed for each material and technique. For acetone dissolution, specimens of 0.5 g of each material were mixed with 10 mL of acetone (electronic grade, Fisher Chemical, Fair Lawn, NJ, USA) in a test tube (Pyrex 50 mL, Fisher Scientific, Hanover Park, IL, USA). All tubes were weighed initially empty and then weighed again following loading with the 0.5-g specimen. Tubes were covered with aluminum foil to prevent light exposure. All specimens initially were agitated (Maxi-mix 1, Thermolyne, Dubuque, IA, USA) until all solid was dissolved and verified visually. Agitation continued with a gyratory shaker (G10, New Brunswick Scientific, New Brunswick, NJ, USA) within a controlled temperature chamber (Norlake Scientific, Hudson, WI, USA) at 37°C for 1 hour. Specimen tubes were centrifuged (Centrifuge 5810R, Eppendorf, Hamburg, Germany) for 15 minutes at 4000 rpm and then decanted twice (5 mL pipette, Novamed Inc, Lawndale Skokie, IL, USA) for a total of 9.5 mL. The specimens were left to dry overnight in the temperature-controlled chamber to ensure all acetone evaporated, and weights of the specimens were measured to the nearest 0.001 g the following morning. The entire process was repeated twice to

Table 1: *Material Specifications as Reported by Manufacturer.*

Brand Name	Filler Type	Manufacturer	Batch Number
IPS Empress Direct	Nanohybrid	Ivoclar	M68450; N10129
Filtek LS	Microhybrid	3M ESPE	N182605; N205729
Kalore	Nanohybrid	GC America	0907141; 0907101
Aelite LS	Microhybrid	Bisco	1000007473; 1000011945

ensure dissolution of the organic matrix. The calculation of filler percentage by weight was the same as the ashing technique except that residual material might include prepolymerized fillers, clusters, and possible silane contents. The formula to determine percentage by weight of the specimens after acetone dissolution was as follows:

$$\text{Weight (Wt) percent} = \frac{\text{Wt after dissolution} - \text{Wt of tube}}{\text{Initial Wt of sample}} \times 100.$$

For the ashing in air, the filler content of the selected composites was determined by using a burn out furnace (Neymatic 101, JM Ney Company, Bloomfield, CT, USA). Crucibles were weighed empty and then weighed after specimen loading using an analytical scale (Analytical Standard, AS200-S, O'Haus, Florham Park, NJ, USA). The crucibles (High alumina, 10 mL, Cole-Palmer Instrument Co, Golden, CO, USA) loaded with specimens were introduced in the furnace after the temperature had reached 650°C and left for 30 minutes. The crucibles with the ashed specimens were again weighed on an analytical scale.

Filler percentages from ashing in air and acetone dissolution technique were statistically analyzed by one-way analysis of variance (ANOVA), independent *t*-tests, Tukey multiple comparison, and Pearson correlation.

EDS is a microanalytic technique that is based on the characteristic x-ray peaks that are generated when the high-energy beam of the electron microscope interacts with the specimen. Each element yields a characteristic spectral fingerprint that may be used to identify the presence of that element within the sample. The relative intensities of the spectral peaks may be used to determine the relative concentrations of each element in the specimen. The x-ray signal is detected by a solid-state silicon-lithium detector, and the construction and efficiency of this detector sets a lower limit on the atomic number that may be detected. Generally, elements heavier than carbon are detectable.[22]

For the SEM evaluation, the residual fillers from ashing in air were mixed with acetone to produce a suspension that was placed on an aluminum SEM stub and allowed to dry. Morphologic and size evaluations were done using a FEI Quanta 200 3D focused ion beam workstation and environmental scanning electron microscope with an EDS system at magnifications of 20,000× and 40,000× at 20-30 kV beam acceleration voltage. The same areas of the samples utilized for SEM observation were additionally x-ray scanned using the same microscope for qualitative and semi-quantitative analyses to determine the elemental distribution profile.

RESULTS

Table 2 shows the means and standard deviations of the materials evaluated by ashing in air and acetone dissolution techniques. Weight percentage of inorganic fillers from ashed specimens ranged from 70.62% to 86.44%. Weight percentage of fillers from acetone dilution specimens ranged between 75.56% and 85.05%. Mean values for each material were significantly different when comparing ashing with acetone dissolution techniques. Both techniques were extremely precise, with standard deviations between 0.1% and 1.1%. Aelite LS and Filtek LS

Table 2: *Filler by Weight (%)*

Product	Manufacturer Values	After Ashing in Air	Manufacturer Difference (%)	After Two Acetone Dissolutions	Manufacturer Difference (%)	Significance (Independent *t*-test)
IPS Empress Direct	79	72.12 (0.1)	9	78.88 (0.5)	0	$p<0.001$
Filtek LS	76	77.86 (0.1)	2	75.56 (0.2)	1	$p<0.001$
Kalore	82	70.62 (0.3)	14	77.73 (0.3)	5	$p<0.001$
Aelite LS	88	86.44 (1.1)	2	85.05 (0.5)	3	$p=0.003$

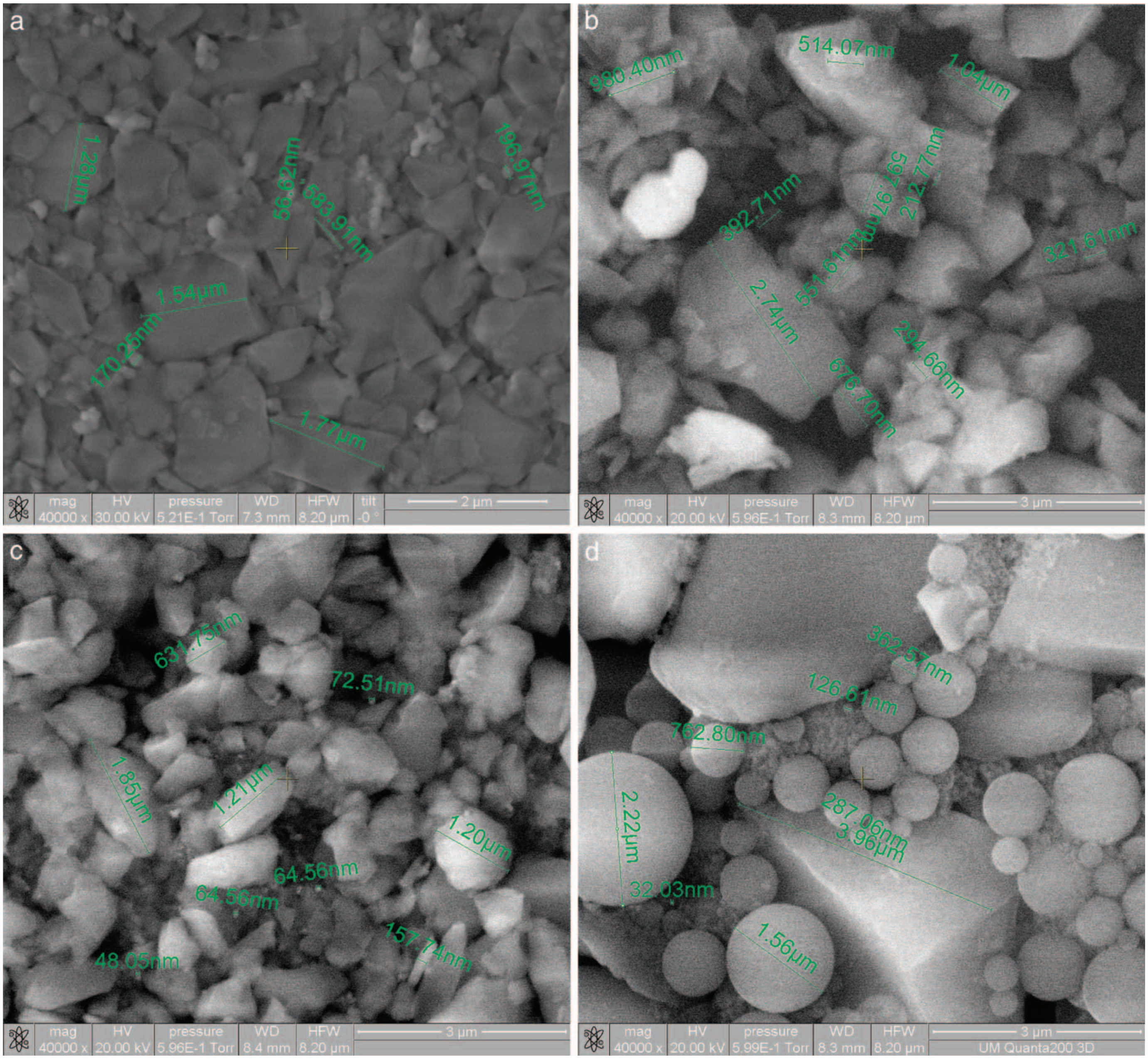

Figure 1. *(A): SEM photomicrographs and filler measurements of IPS Empress Direct composite after acetone dissolution at 40,000× magnification. (B): SEM photomicrographs and filler measurements of Filtek LS composite after acetone dissolution at 40,000× magnification. (C): SEM photomicrographs and filler measurements of Kalore composite after acetone dissolution at 40,000× magnification. (D): SEM photomicrographs and filler measurements of Aelite LS composite after acetone dissolution at 40,000× magnification.*

showed closer results for percentages by weight to manufacturers' reported data when using ashing (2%) and acetone technique (1%-3%). On the other hand, Kalore and IPS Empress Direct showed results closer to manufacturer reported data when using acetone dilution for weight measurements (0%-5%). Aelite LS had a significantly higher loading than the others for weight percentage for both ashing in air and acetone dilution. Both techniques were highly correlated (r=0.72).

Representative SEM photomicrographs (backscattered electron images) of the composites evaluated in this study at 40,000× are shown in Figure 1A through D. Irregular particles (3.96 µm) in conjunction with smaller spherical (32.03 nm-2.22 µm) filler particles and agglomerated particles were observed in Aelite LS. Overall, Aelite LS was the material that

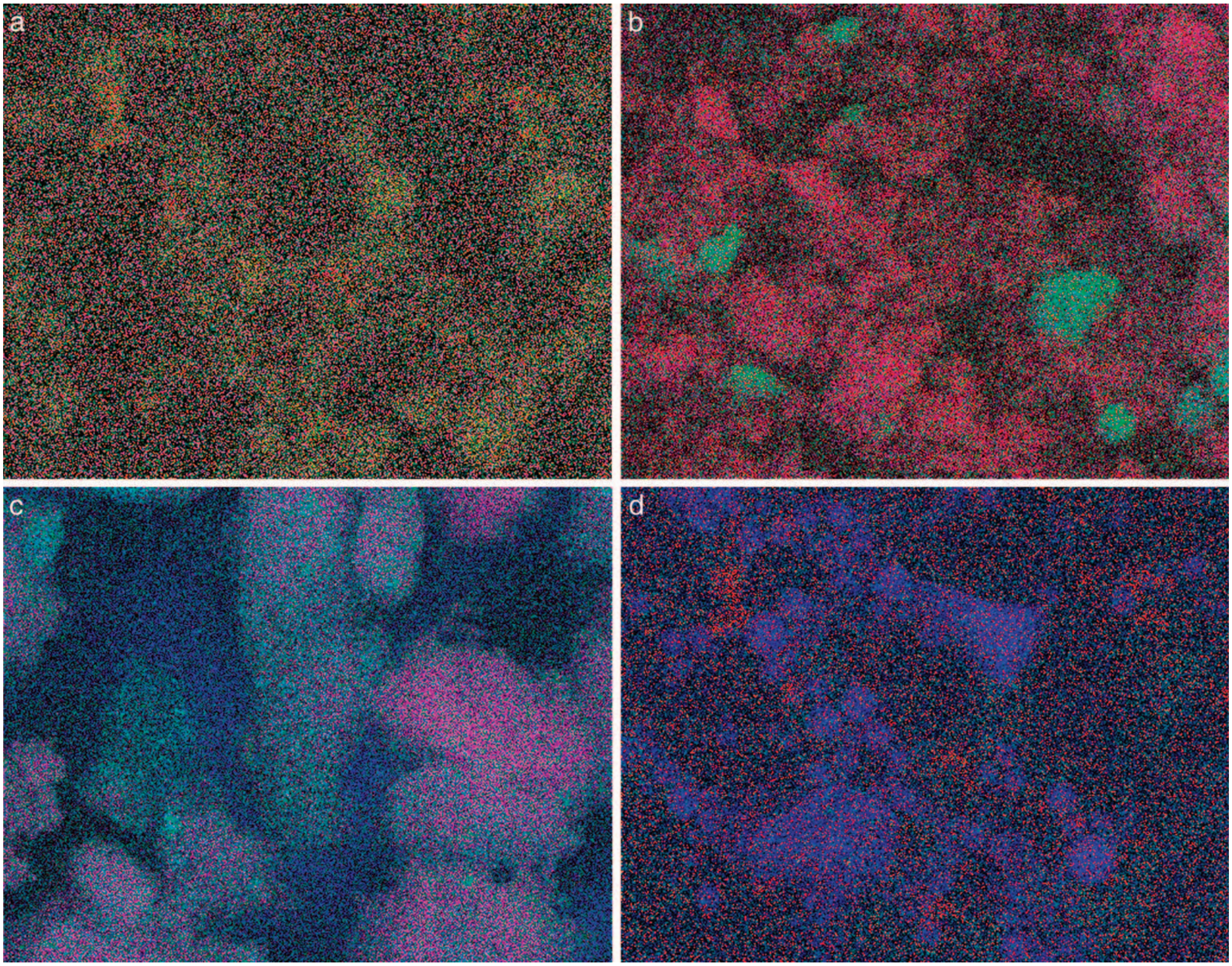

Figure 2 (A): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the IPS Empress Direct composite: Si-yellow, Al-pink, O-purple, F-green, Yb-red, Ba-light blue. (B): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the Filtek LS composite: Si-pink, Mg-blue, Al-purple, F-green, O-red, Y-light blue. (C): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the Kalore composite: Si-pink, Al-purple, Mg-blue, F-green, O-yellow, Yb-light blue. (D): SEM photomicrographs used for EDS analysis and color mapping obtained by the superimposition of every individual element distribution in the Aelite LS composite: Si-blue, Al-red, O-light blue.

showed the biggest (3.96 μm) and smallest filler particles (32.03 nm) (Figure 1D). Irregular and splintered filler particles of different sizes were seen in Filtek LS, IPS Empress Direct, and Kalore. In Filtek LS, most of these particles were somewhat larger (598 nm-2.74 μm), while the remaining particles were between 212.77 nm and 321.61 nm (Figure 1B). Kalore showed the most regularly shaped filler particles among all composites evaluated, with a mix of medium (631 nm-1.85 μm) and small (48 nm-157 nm) size particles (Figure 1C). On the other hand, in IPS Empress Direct, a more irregular shape pattern in the filler particles was observed, with particles typically ranging from 1.28 μm to 1.77 μm, and the smallest particles being around 56.62 nm (Figure 1A). The elements detected in the resin composites are shown in Figure 2A through D and Table 3. Silica, aluminum, and oxygen were found in all of the resin composites tested and were the main components of the fillers.

DISCUSSION

Many methods could be used to study the filler concentration by weight. Thermogravimetry is a technique in which the mass of a material is

Table 3: *Elements Detected and Filler Content of Resin-based Composites*

Product	Filler Particle Size Range	Elements Detected
IPS Empress Direct	57 nm-1.8 μm	Si, Al, O, F, Yb, Ba
Filtek LS	212 nm-2.7 μm	Si, Al, O, Mg, F, Y
Kalore	48 nm-1.9 μm	Si, Al, O, Mg, F, Yb
Aelite LS	32 nm-4 μm	Si, Al, O

Abbreviations: Aluminum (Al); barium (Ba); fluorine (F); magnesium (Mg); oxygen (O); silica (Si); strontium (Sr); ytterbium (Yb); yttrium (Y); and zirconium (Zr).

monitored as a function of temperature and time as a sample specimen is subjected to a controlled temperature program.[11,21,26] The thermogravimetry curve pattern varies according to the materials tested, thus reflecting the variations in organic composition. Ashing in air, on the other hand, is the technique most frequently used to determine the percentage of fillers by weight. It is based on the elimination of the organic fraction of the resin composite by heating the composite to a constant temperature. Some authors ashed the materials in an electric furnace at temperatures that ranged between 600° and 700°C, respectively, for 30 minutes.[3,8-10] Aelite LS (86.44%) and Filtek LS (77.86%) composites showed the highest amounts of inorganic content after ashing in the furnace. When comparing these results with the manufacturer's reported data, the microhybrid materials showed closer results for ashing in air (2%). On the other hand, the nanohybrid composite's (Kalore and IPS Empress Direct) results after ashing showed a greater difference from the manufacturer's data (9%-14%).

Variations within these composites could be explained by the definition of filler content. The filler content reported by manufacturers sometimes includes only the inorganic filler particles, and anything organic is vaporized when the composite is subjected to ashing temperatures. Prepolymerized particles in many composites use the organic matrix and inorganic filler particles, which are first cured into solid blocks and then milled or ground down into sizes ranging from 17 μm to 60 μm.[1,27] The milled particles are added to a nonpolymerized resin along with inorganic particles and dispersed aggregates to increase loading. Prepolymerized fillers are relatively large fillers with less surface area, enabling greater weight filler loading and thereby resulting in less volumetric shrinkage. These larger fillers also prevent the resin matrix from moving as a result of friction between the resin and the prepolymerized filler surface during curing, thereby reducing shrinkage.[28]

Prepolymerized particles present different shapes and sizes, and they can have as much as 50% organic content.[10,11,21,22,24,28,29] It is of interest as to what constitutes filler content with different manufacturers. IPS Empress Direct and Kalore have prepolymerized particles in their filler composition. During ashing in air, organic matrix as well as prepolymerized particles can be vaporized, which can result in significantly lower percentage by weight values (Table 2). Prepolymerized particles with organic content were included in the manufacturer filler calculation of IPS Empress Direct and Kalore, which is reflected using the acetone dissolution technique. The manufacturer of IPS Empress Direct reported 50.2% barium-aluminum-fluorosilicate glass, 9.8% ytterbium trifluoride, and 19.6% prepolymerized particles in its total filler percentage by weight of 79%. On the other hand, Kalore reported a higher filler content, with a total of approximately 50% fluoroaluminosilicate and strontium glass and 32% prepolymerized particles. So, Kalore and IPS Empress Direct are directly related in how their filler percentages by weight were calculated. These nanohybrid composite results after acetone dissolution were closer to the filler weight reported by the manufacturers, and the small differences may be due to some particles lost during each acetone pipette decantation step. However, these small differences were not seen when using ashing in air technique instead of acetone for the same measurement. According to their manufacturer, Aelite LS and Filtek LS do not have prepolymerized particles incorporated in their filler composition, which resulted in the smaller difference between ashing in air and acetone dissolution (less than 3%). The microhybrid manufacturers' reports were very close to the results obtained in this study, and the small differences could be due to loss of some particles during handling of samples from furnace to analytical scale after ashing or loss of particles during decantation in the acetone dissolution. Aelite LS had the highest percentage of filler content by weight for both techniques, while Filtek LS had the lowest percentage of filler by weight after acetone dissolution (75.56%).

Others factors could explain differences found between our data and those given by the manufacturers. The first one is the variable amount of silane. The silanation process plays a main role in the adhesion of the organic resin matrix to the inorganic mineral fillers.[30] Manufacturers and laboratories treat the filler-matrix interface according to their own methods and use different ways to calculate the percentages of fillers.[8] Some manufacturers seem to evaluate the percentage of fillers by weight before the silanization process of the fillers, while others include the percentage of silane coating in their calculation. In addition, the surface area of the fillers will affect the percentage of silane used—the smaller the fillers, the higher the quantity of silane.

Furthermore, samples from each composite were dissolved in acetone to evaluate the filler content by weight using a technique that preserves the prepolymerized particles. Previous studies had evaluated filler structure and size,[8,10,11,22,24] but only one previous study has determined filler percentage by weight using the acetone dissolution technique.[17] With the new generation of composites, it will be necessary to evaluate these materials considering their prepolymerized particles. A pilot study was done to determine the amount of cycles of acetone dissolution necessary to obtain a stable percentage by weight. No significant differences were found between the second and third dissolution; therefore, two cycles of acetone dissolution were chosen as a standard procedure. Kalore showed the highest difference in filler content by weight for the acetone dissolution technique (77.73%). A similar increase was seen with IPS Empress Direct when comparing ashing in air (72.17%) and acetone dissolution (78.88%). Aelite LS and Filtek LS showed a decreased weight percentage when using acetone dissolution, which may be related to residual nanoparticles suspended in the acetone after centrifuge sedimentation that are lost during pipette aspiration.

For the filler morphology analysis, the samples dissolved in acetone were collected and dissolved a third time for mounting purposes. On the SEM evaluation, all composites showed an irregular to splintered shape except for Aelite LS, which is the only composite in this study that contains spherical particles mixed with irregular-shaped particles. A spherical shape is known to have many advantages such as to allow an increased filler load for composites and also to enhance their fracture strength, surface roughness, and shrinkage strain since mechanical stresses tend to concentrate on the angles and protuberances of the filler particles.[3,8,10,31,32] The spherical particles had different diameters that ranged from 32 nm to 2 μm, while the bigger irregularly shaped particles presented an average size of 4 μm with some nanoclusters and dispersed nanoparticles surrounding the bigger filler particles.

For Filtek LS, the SEM images confirmed a distinct filler morphology of irregularly shaped fillers. The manufacturer of Filtek LS claimed that the radical change in the shape of the fillers, among the Filtek composite varieties, is related to the specific characteristic that the silorane-based organic matrix needs in its composition. Silorane is known to contain quartz particles, which cannot be processed by a sol-gel technique and may explain the more irregular morphology compared with other materials provided by the same manufacturer. According to the manufacturer, the average size range was from 40 nm to 1700 nm; however, the SEM images in this study showed particles that ranged from 200 nm to 3 μm in size. In addition, the images showed a more heterogeneous size pattern when compared to Kalore, the other microhybrid material.

According to the manufacturer, Kalore contains 60% filler content by weight of 400 nm nano-sized modified strontium glass and 20% filler content by weight of 100 nm of lanthanoid fluoride. Prepolymerized nanoclusters of fillers, inorganic fillers, and mono-dispersed particles, contained in this specific nanohybrid composite present size ranges between 16 nm and 17 μm. When the samples were evaluated under SEM, the images showed more regular shaped filler particles whose bigger particles ranged from 632 nm to 1.8 μm and small particles ranged from 48 nm to 158 nm. Among all materials, Aelite LS (32 nm) and Kalore (48 nm) are the materials that had the smallest filler particles in their structure. In addition to Kalore and Fitek LS, IPS Empress Direct also showed irregular shaped particles with large fillers that ranged from 1.28 μm to 1.77 μm and smaller aggregates of particles with sizes from 56.62 nm to 196.97 nm.

In this study, prepolymerized particles were not evaluated for elemental analysis because the components of each composite material were disrupted during the acetone dissolution technique; therefore, further studies should be done keeping the organic matrix of each resin-based composite.

Many modifications have been made recently to the elemental composition of filler particles. Elements such as aluminum and lithium were added to decrease the hardness on these particles, to be able to break them in smaller sizes. Barium, yttrium, zinc, and strontium are added to develop radiopacity. Some manufacturers have claimed that yttrium fluoride can be incorporated to release fluoride ions for anticaries activity.[21] In this study, the EDS analysis of the fillers revealed a variety of elements, depending upon the individual products. Colors representing each element were picked randomly for each analysis (Table 3, Figure 2).

For IPS Empress Direct (Figures 1A and 2A), the filler particle sizes are well dispersed, and the predominant elements of silica (yellow) and fluoride (green) appear to be concentrated in the larger particles; ytterbium (red) and barium (light blue), however, appear to be in smaller, more dispersed particles. Aluminum is generalized throughout the field, in keeping with the stud background on which the fillers were placed.

For Filtek LS (Figures 1B and 2B), the particles are larger and more distinct (Figure 1B). Silica (pink) and oxygen (red) are heavily concentrated in the more distinct larger particles (Figure 2B), and separate less numerous particles contain a high concentration of fluoride (green) as well as magnesium (blue) and yttrium (light blue) for radiopacity. Again, the aluminum (purple) forms the general background color from the mounting stub.

For Kalore (Figures 1C and 2C), the larger particles appear to contain heavier concentrations of ytterbium (light blue) and magnesium (blue) with some aluminum (purple) microconcentrations within specific particles. Other particles appear to contain heavier concentrations of silica (pink). Small amounts of fluoride (green) were distributed around the edges of most particles, and there was no oxygen (yellow) evident in most fields.

For Aelite LS (Figures 1D and 2D), the predominant element is silica (blue) in both larger and smaller particles, and small concentrations of aluminum (red) in smaller particles, with a general distribution of oxygen (light blue) in low concentrations. There was no evidence of fluoride, magnesium, or a heavy metal ion.

A clinician selects a composite expecting good physical properties and esthetics. If that decision is made based on the manufacturer's reporting of filler weight percentage, the clinician may be misled by assuming that those percentages are inorganic content. This study has shown that the filler weight percentage that is reported may be affected by techniques used to determine that percentage as well as by the inclusion of prepolymerized particles.

CONCLUSIONS

1. Mean values for all materials were significantly different for both ashing and acetone dissolution techniques, but there was a strong correlation between the two tests.
2. Aelite LS had significantly higher filler content for both ashing and acetone dissolution.
3. Kalore had significantly lower filler content for ashing (70.62%), and Filtek LS had significantly lower filler content for acetone dissolution (75.56%).
4. Manufacturer reported filler content for Aelite LS (88%) and Filtek LS (76%) approximated the study results for both techniques, while Kalore (82%) and IPS Empress Direct (79%) were only similar for acetone dissolution, indicating higher content of prepolymerized particles.
5. Morphologic examination showed spherical shaped particles for Aelite LS and splintered and irregular shaped particles for the other materials.
6. The elements detected were silica (Si), aluminum (Al), strontium (Sr), yttrium (Y), magnesium (Mg), fluorine (F), ytterbium (Yb), oxygen (O), zirconium (Zr), and barium (Ba). Si, Al, and O were the common elements in the composition of all four resin composites evaluated.
7. Manufacturers should provide two filler content values for a composite resin material—inorganic filler content and total filler content.

Conflict of Interest

The authors of this manuscript certify that they have no proprietary, financial, or other personal interest of any nature or kind in any product, service, and/or company that is presented in this article.

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