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TECHNICAL NOTE

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Detection and Classification of Composite Resins in Incinerated Teeth for Forensic Purposes

ABSTRACT: The great demand for esthetic restorations has resulted in placement of large numbers of composite resin fillings. The popularity of these materials is reflected in the quantity and variety of resin brands currently on the market. The ability to distinguish resin brands can aid in positive identification of burn victims, assuming that appropriate dental records exist. Scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) was used to characterize the structure and composition of 10 modern resins. The structure of each resin was unique to manufacturer, and elemental analysis allowed separation into distinct groups. These 10 resins were also placed in extracted teeth and incinerated at 900°C for 30 min, simulating near cremation conditions. The resins were identifiable by SEM/EDS after incineration, and the elemental composition remained almost unchanged. The data produced are immediately useful for resin identification in forensics, and comparative analysis can be readily performed using standard equipment. This work represents the initial stage of database generation.

KEYWORDS: forensic science, forensic odontology, restorative composite resins, SEM/EDS, incineration

Determining the identity of a victim that has sustained extensive damage to the tissues because of burning is a challenging task. When that victim has been incinerated to the point where only a few fragments of teeth and bone remain, that task may become impossible. Teeth are very resistant to high temperatures and remain recognizable even after prolonged exposures to heat. The human dentition, with wear, alignment, and combinations of missing, restored and unrestored teeth is as unique and individual as a fingerprint. This specific nature creates a gold standard for victim identification and can be applied even in cases when only a single tooth exists. Thus, any method that gives additional information such as the brand of restorative material is valuable and enhances the possibility of positive identification (1–4). Therefore, it is important to be able to distinguish charred restorative materials from charred tooth structure, and it is even more important to identify specifically those restorative materials by brand name.

In a 1999 murder case, a single tooth was retrieved from a burn pit. The tooth was analyzed and found to have an amalgam restoration bonded into place with a newly introduced cement. The cement was found to contain zirconium and silicon, consistent with Rely X ARC, produced by 3M ESPE (St. Paul, MN). The restoration matched the dental records for the missing person and this analysis helped in positive identification of the victim (5).

There has been a dramatic increase in the demand for esthetic restorations by the public. Tooth-colored resin composites are being placed in large numbers. In response to this demand, manufacturers have developed over 50 resins available for use by dentists (6). The increased use of resin has presented a challenge to both dentist and forensic odontologist alike, as well-placed resins can be difficult to recognize both clinically and radiographically. During postmortem examinations various methods have been utilized for enhanced gross identification of the presence of these restorative materials. These include plaque disclosing solution, dyes (alizarin red), trans-illumination, and quantitative light-induced fluorescence (7,8). Incineration further complicates the gross or macro-identification process. Once these restorations have been identified, further analysis of the material is possible.

Composite resin consists of an organic resin matrix surrounding inorganic filler particles. Many of the resins contain filler particles of unique elemental composition and size for reasons such as radio-opacity, handling, and polishing characteristics.

Fortunately, for identification purposes, the resin fillers are recognizable by elemental composition and can be detected in samples by scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) analysis. It is also the filler particles that are resistant to high temperatures and which remain detectable by SEM/EDS. EDS analysis produces an X-ray spectrum that identifies inorganic elements and their relative concentrations. The use of backscattered electron imaging (BEI) with the SEM allows determination of filler particle size and distribution. It was the goal of this research to assess the structure and elemental compositions of resins currently on the market and to determine if these remain identifiable after exposure to extreme temperatures.

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TABLE 1—*Resins used in this study and descriptions of their content as provided by the manufacturer.*

Filtek Supreme Plus (3M ESPE)
SiO_2 Nanosilica filler, ZrO_2/SiO_2 nanoclusters
QUIXX (Dentsply, Milford, DE)
Strontium aluminum sodium fluoride phosphate silicate glass
Gradia Direct (GC America, Alsip, IL)
Silica, prepolymerized fillers, fluoro-alumino-silicate glass (posterior only)
Heliomolar (Ivoclar)
Dispersed silica-silanized copolymer, ytterbium triflouride
Tetric Ceram (Ivoclar, Amherst, NY)
Barium glass, ytterbium triflouride, Ba–Al floursilicate, silicon dioxide
4 Seasons (Ivoclar)
Barium glass, ytterbium triflouride, Ba–Al floursilicate, silicon dioxide
TPH3 Micro Matrix Restorative (Dentsply)
Barium fluoro aluminum boro silicate glass, fumed silica
Point 4 (Kerr)
Barium aluminoborosilicate
Premise (Kerr)
Prepolymerised filler, barium glass, silica nanoparticles
Venus (Heraeus Kulzer)
Barium aluminum boron flouride silica glass, dispersed silicon dioxide

The condition of the teeth and the dental materials recovered from burn victims is dependent on the maximum temperature the fire has achieved as well as how long it has burned. A body will be completely destroyed, cremated, at 870–980°C for $1-1\frac{1}{2}$ hours (9). Even at this extreme, tooth fragments and restorative materials can remain (10–13). When a tooth is incinerated, dehydration causes shrinkage and fragmentation of the tooth causing displacement of the restorative material. Physical changes can be extensive (14–17). Both tooth structure and resins can range in color from chalky white to blackish, making visual identification from fragments difficult. The use of BEI and EDS analysis with the SEM, however, allows one to distinguish resin from tooth structure in charred mixtures and to identify elemental composition. In this study, the structure and elemental compositions of 10 modern resins were analyzed, first to determine if it was possible to distinguish brands, and second whether elemental composition can be confirmed after incineration.

SEM/EDS instrumentation is commonly available and comparative analysis can easily be performed. The data produced in this study are reproducible and allow formation of a database. This database could be referenced as needed to make comparisons of an unknown resin material to a known reference restorative material (18–20).

Materials and Methods

Ten disks of resin material, 1 cm in diameter, were prepared and cured according to manufacturers instructions. Table 1 lists the resins used in this study as well as the compositions described

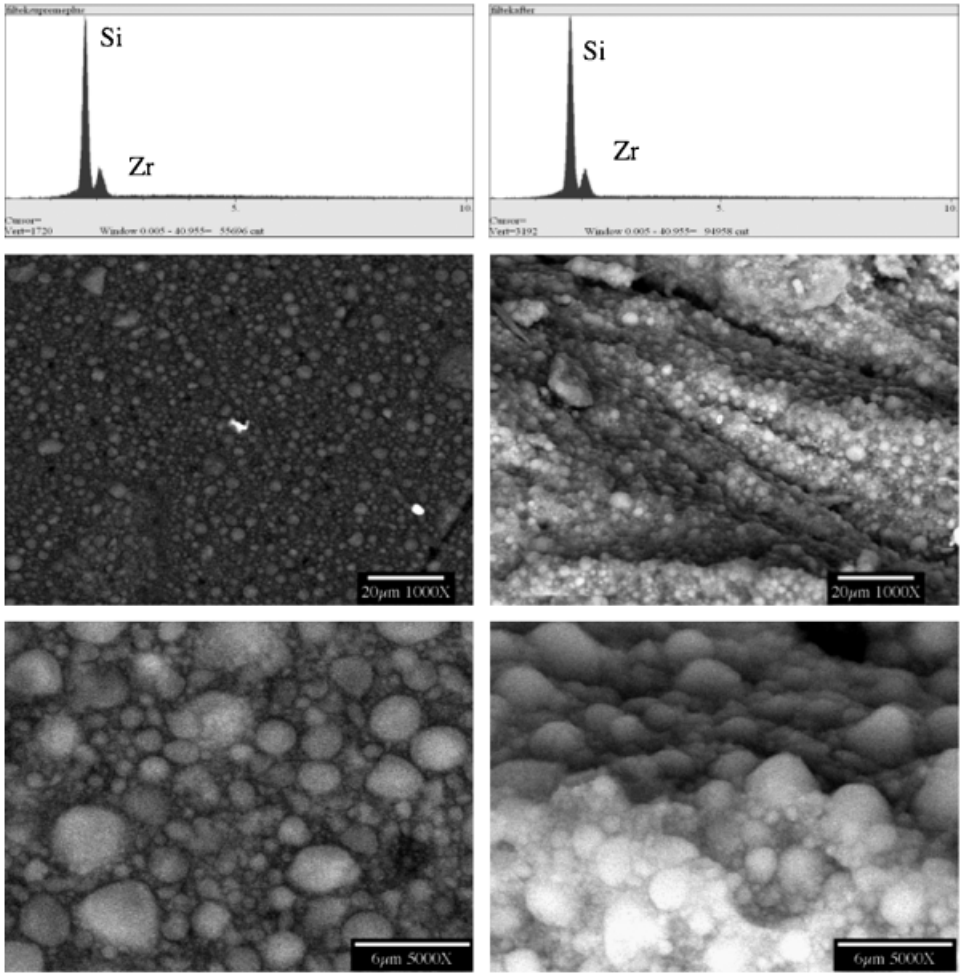

FIG. 1—*Filtek Supreme Plus (3M), universal nanofilled resin. Energy dispersive X-ray spectrum and backscattered electron imaging images before (left) and after incineration (right). The spherical agglomerates of zirconium silicate are still recognizable after incineration.*

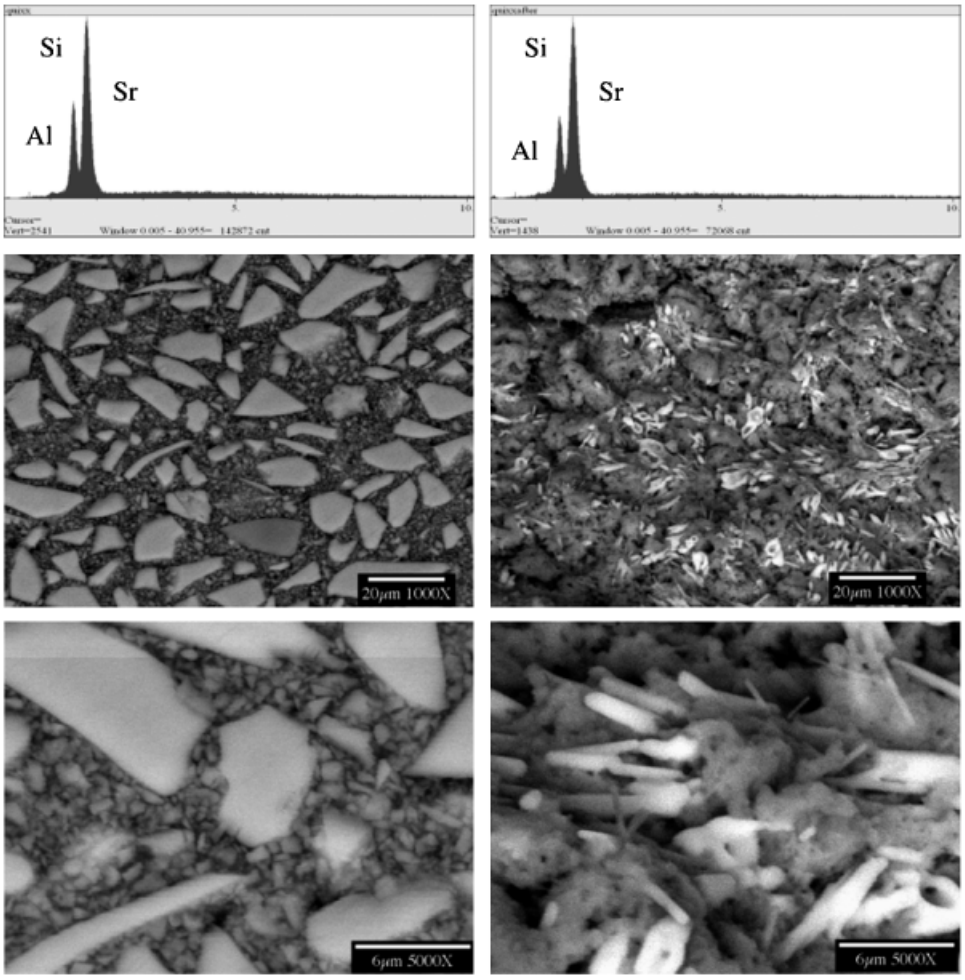

FIG. 2—*Quixx (Dentsply) posterior resin. Energy dispersive X-ray spectrum and backscattered electron imaging images before (left) and after incineration (right). Large particles of aluminum strontium silicate. After heating, a strontium-rich phase separated (bright regions, right).*

according to the manufacturer (5). Manufacturer addresses can also be found in this reference. Four separate areas on each disk were analyzed and the average of elemental composition was taken. Table 3 lists the averaged elemental composition as detected by EDS. These disks represented the control samples for analysis of structure and composition before incineration.

The same resins were also placed in extracted teeth. The teeth were prepared with mesial and distal preparations. The preparations were etched with 37% phosphoric acid for 15s, rinsed, and gently dried with an air syringe until the dentin showed a moist appearance. Prime and Bond NT bonding agent was next applied, gently air-dried, and cured for 20s. Each mesial and distal preparation received a different resin. The resins were then cured for 30s. The filled teeth were placed in a burnout oven and heated at 900°C for 30 min in air in a ceramic crucible. These conditions represent an extreme. In typical cremation situations the teeth would be protected by soft tissue and bone, requiring $1-1\frac{1}{2}$ h to reduce them to fragments (8). Because the extracted teeth were not protected, they reduced to fragments in a much shorter time.

After removal from the oven, the remains were inspected using a stereomicroscope to first determine if resin material could be distinguished visually from tooth structure and second, to retrieve presumed resin from the fragmented debris. Next, the debris was analyzed by SEM/EDS.

EDS Analysis

Both the control sample disks and the incinerated materials were analyzed under the following conditions: × 500 magnification 25 keV acceleration voltage, 43° takeoff angle, for 100 s live time. The low magnification of × 500 was selected because the heterogeneous structure of the resins required a large field to obtain a correct average composition. A spectrum was obtained and the elements were identified. Semiquantitative standard-less analysis was performed from the spectra to derive atomic percent concentrations of the elements. Four separate fields were analyzed and an average of the four reported. In this study, a beryllium (Be) window detector was used making detection of elements below sodium (Na) in the periodic table not possible. These analysis conditions are typical for many SEM/EDS installations.

Electron Imaging

Most published SEM images are secondary electron images, in which the contrast is dictated by surface morphology. Secondary electron images often contain microstructural information with very high resolution (see dentin picture in Fig. 6). In backscattered electron images, however, the contrast in the pictures is dictated by average atomic number; thus, bright regions in the images represent areas of high atomic number. Areas of different gray

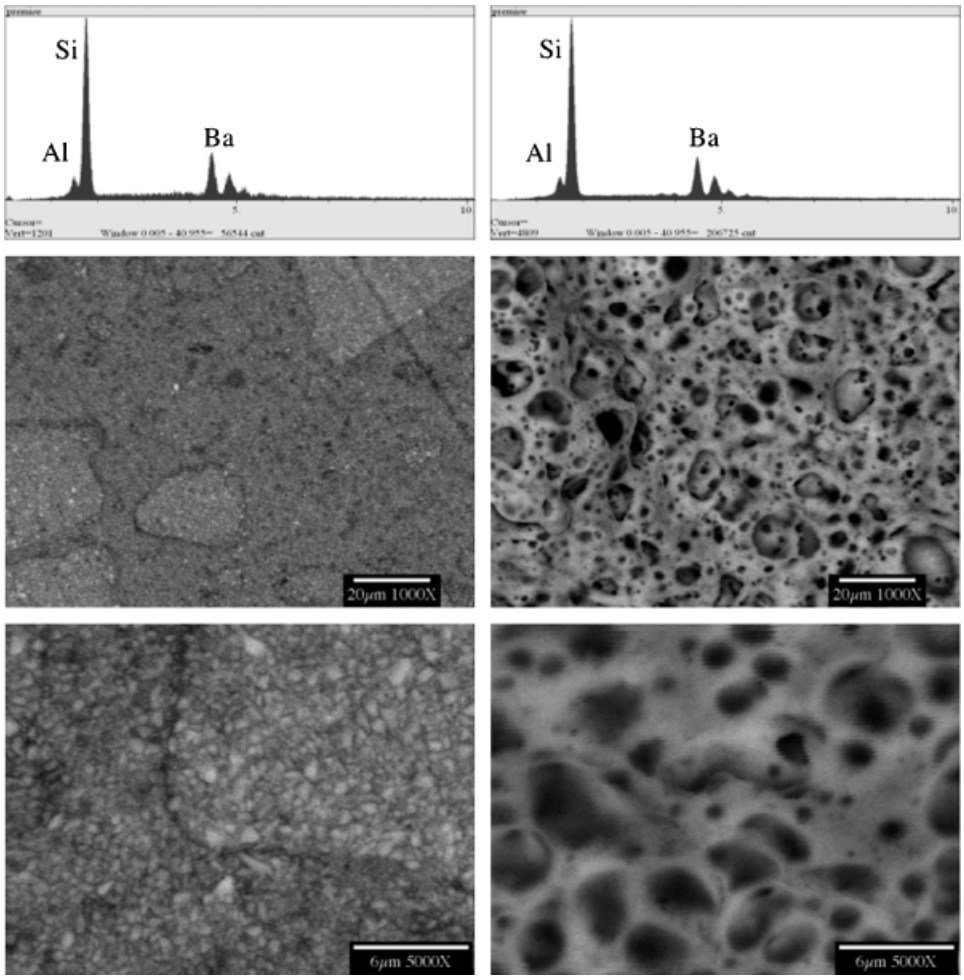

FIG. 3—*Premise (Kerr), universal nanofilled resin Energy dispersive X-ray spectrum and backscattered electron imaging images before (left) and after incineration (right). Large particles of prepolymerized barium glass-filled composite in a matrix containing the same type of filler particle.*

level in the images, therefore, indicate areas of different chemical phases. BEI of the control and incinerated resins were taken at × 1000 and × 5000. These magnifications were considered adequate to characterize typical microstructure and particle size.

Results

Preincineration

The control resin samples each had a unique microstructure. The images taken at × 1000 and × 5000 reflect the heterogeneous, but typical microstructural characteristics of each resin. These showed different particle sizes and filler constituents distinct to each resin. Based on the structure alone, it was possible to distinguish all brands of resin used in this study. Examples are shown in Figs. 1–4.

Elemental analysis of the control samples allowed the resins to be divided into six elementally distinct groups as shown in Table 2. Four of the resins apparently use the same inorganic filler glass, aluminum barium borosilicate. The three manufacturers represented by this group describe the filler content as a mixture of silicate (SiO_2) and aluminum barium glass. Careful analysis revealed that one manufacturer mixed different proportions of these fillers. Point 4 and Premise, both Kerr (Orange, CA) products, and Venus (Heraeus Kulzer, Armonk, NY) were elementally indistinguishable by Si/Ba ratio. TPH3 (Dentsply), had a ratio of Si/Ba that was significantly different from the other three. Thus, the group of four could be further subdivided based on the Si/Ba ratio. This further distinction allowed separation into seven groups. Four Seasons and Tetric Ceram, both Ivolcar products, were close enough in elemental composition to be indistinguishable within the limits of the technique.

When elemental composition was combined with micro-structural analysis, all 10 resins could be distinguished and identified by brand name.

A parallel study was performed to determine if there were elemental differences between shades of resins within a brand. No differences were detected.

Postincineration

All incinerated resins underwent a microstructural alteration, also shown in Figs. 1–4. Sintering, fusing, and melting of the filler particles occurred, and the resin matrix was burned off. In the resins with more refractory fillers, remnants of the original microstructure were recognizable (Fig. 1).

After incineration, some of the resins remained partially intact in the prepared tooth. The tooth itself fragmented and underwent local color changes varying from chalky white to black. The resins underwent a similar range of color changes; however, the color

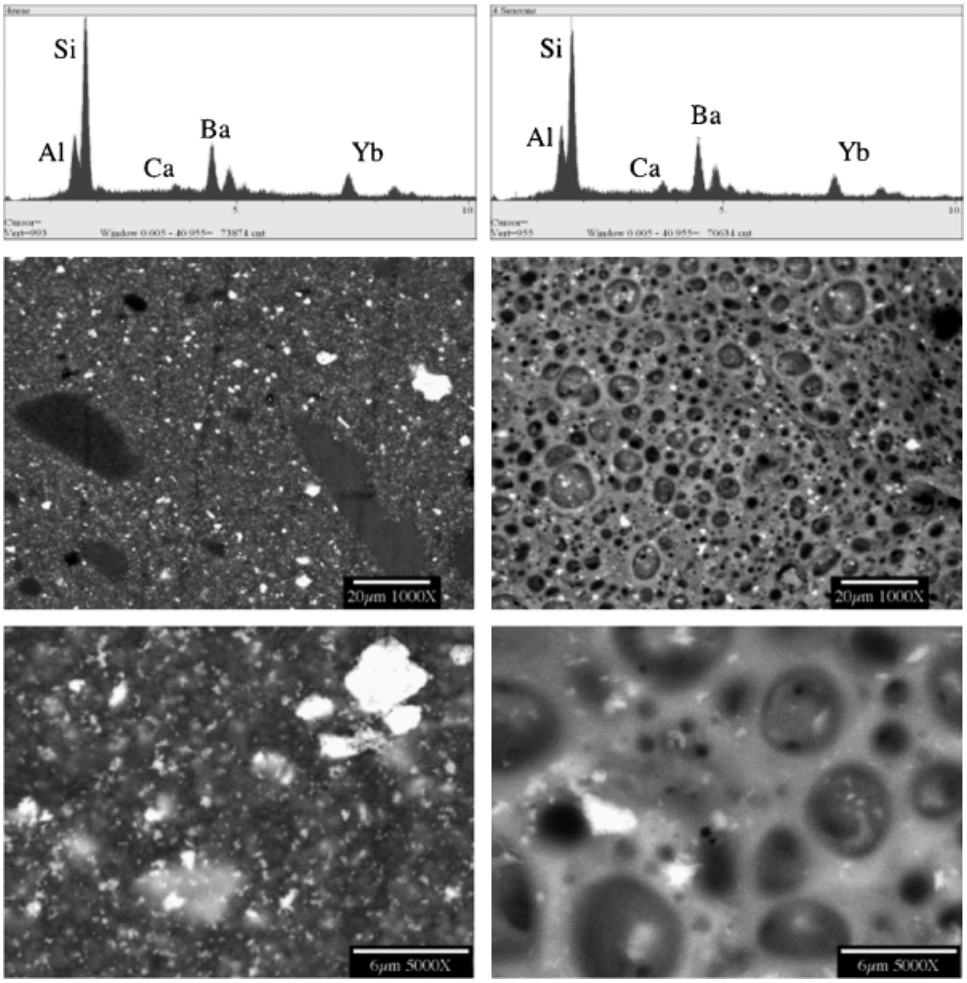

FIG. 4—*4 Seasons (Ivoclar), microhybrid resin. Energy dispersive X-ray spectrum and backscattered electron imaging images before (left) and after incineration (right). Large particles of prepolymerized resin, and large ytterbium glass particles.*

change was unique to brand type (Table 2). Resins that remained in the tooth were easily identifiable visually and with the stereomicroscope.

In some cases, the restorations dislodged from the tooth. The respective color changes made it difficult to distinguish resin from tooth structure by visual inspection using a stereomicroscope.

The same field that was presented under the stereomicroscope was imaged by BEI. This technique was able to readily distinguish and identify tooth structure from resin (Fig. 5). In the BEI images, areas of different elemental composition have different gray scale values and appear as areas of different contrast in the image. Particles appearing lighter or darker than tooth structure were further analyzed by EDS to determine their elemental composition. In Fig. 5, three particles of suspected resin can be seen to be lighter than the surrounding tooth structure.

TABLE 2—*Resins were initially divided into six groups based on unique combinations of elemental composition. This grouping was still valid after incineration.*

Elements Detected	Resin Brand	Color Change
Si, Al, K	Gradia (GC America)	White
Si, Yb	Heliomolar (Ivoclar)	White
Si, Al, Ba, Yb	4 Seasons (Ivoclar)	Gray
	Tetric Ceram (Ivoclar)	Gray
Si, Al, Sr	Quixx (Dentsply)	White
Si, Zr	Filtek (3M ESPE)	White
Si, Al, Ba	Point 4 (Kerr)	Dark gray
	Premise (Kerr)	Dark gray
	TPH3 (Dentsply)	Dark gray
	Venus (Heraeus Kulzer)	Dark gray

Particles in fields of charred remains are readily distinguishable by SEM/EDS. The microstructure of charred tooth particles is uniquely different from charred resin. Figure 6 illustrates the difference between the microstructure of tooth fragments and resin. Interestingly, the tubular structure of the dentin is retained and the apatite re-crystallized forming a globular microstructure. The charred resin had a fused or melted microstructure. Further confirmation of particle origin can be made by EDS.

After incineration, the elemental composition of the resins underwent some changes, but the unique combinations of elements was retained. This allowed each resin to be identified as belonging to one of the six elementally unique groups. The resins belonging to the group containing barium glass, however, could not be identified individually, because of postincineration changes in elemental composition. Before incineration, these resins had unique microstructures that allowed recognition of brand. After incineration these resins underwent similar color changes and became indistinguishable by EDS and structural analysis. Nonetheless, because of the unequivocal nature of the analysis, a resin particle with that composition must belong to that group. Identification

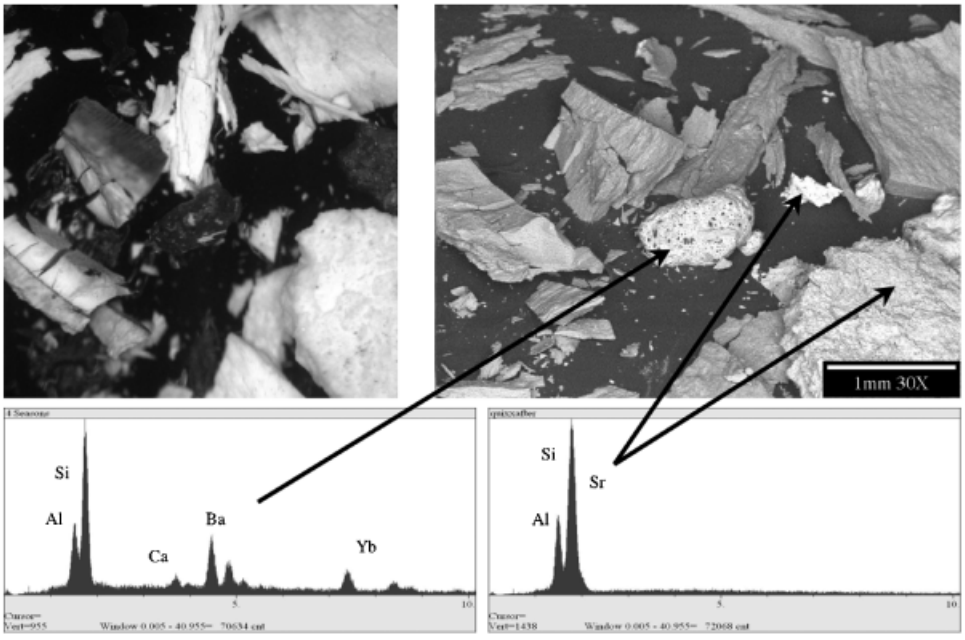

FIG. 5—*Upper left: stereomicroscope view of tooth debris after incineration. Upper right: backscattered electron image of same field of debris showing brighter resin remains. Note the difference in contrast. Lower: Energy dispersive X-ray spectra from the identified particles, revealing that the resins present are Quixx (right) and either 4 Seasons or Tetric (both Ivoclar, left).*

may still be aided by this analysis as the use of other resins is ruled out.

Table 3 lists elemental composition both before and after incineration in atomic percent (normalized to 100%). Use of the detector type described above did not permit detection of the elements B, O and F, which were described as being components of some of the materials. Their detection was not necessary to distinguish the resins and would not have affected the outcome of the analysis.

Discussion

Previous studies have examined various restorative materials from a forensic aspect. Tooth structure, amalgam, and porcelain have been analyzed by SEM. The thermal behavior of some restorative resins has been studied, and there have been case reports of the use of SEM to gain further information for positive identification (1–5,10–13). To our knowledge, however, no systematic attempt has been made to produce an analytical survey of restorative materials with this technique.

The results of this study are of immediate utility in identifying restorative resins for forensic purposes. Both structural characteristics and elemental composition have been described for 10 modern resins. The resins are individually and unequivocally distinguishable by a combination of structure and elemental composition when not subjected to high temperature. This may also be a useful finding in cases where remains are unrecognizable because of advanced decomposition or skeletonization, and composite resin restorations are present. It may be applied to any circumstance or situation where a composite restorative material needs to be classified and identified.

Furthermore, the effect of high temperature on resin composition has been shown to have no negative effect on resin identification by elemental analysis, and it was still possible to unequivocally separate the 10 resins into six groups. Thus, in situations such as airline crashes, explosions, or wrongful cremation, there exists the possibility of positive identification through resin

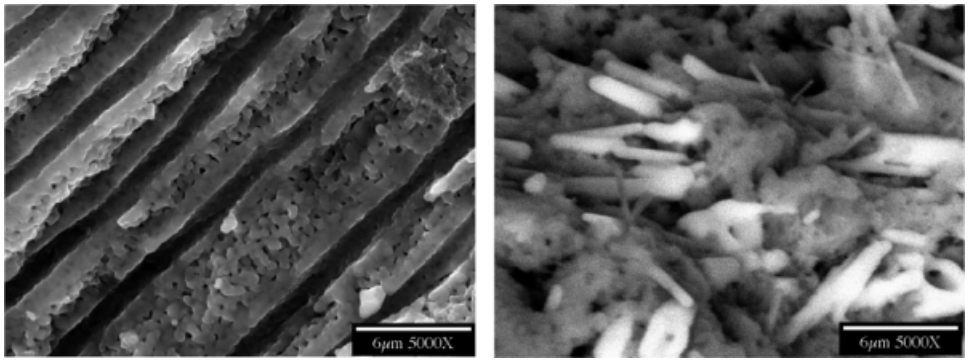

FIG. 6—*Secondary electron image of charred dentin, left. The dentin tubule structure is retained, but the apatite has undergone recrystallization, resulting in a globular structure. By comparison, the structure of a charred resin (Quixx), right, is distinctly different.*

TABLE 3—*Elemental composition of resins by energy dispersive X-ray spectroscopy in atomic percent, before and after incineration.*

Elements, at%	Filtek		Heliomolar		Quixx		Gradia		TPH3		Point 4		Premise		Venus		4 Seasons		Tetric	
	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After
Na					1.1															
Al					27.1	25.3	6.9	8.2	8.3	8.5	8.4	8.8	6.6	7.8	8	7.1	6	7.3	7.7	4.7
Si	80.4	79.9	86.8	88.2	38.5	33.9	80.8	82.2	63.8	72.7	74.6	76.5	75.4	75.1	71.9	71	63.6	61.2	67	65
K							12.3	9.6												
Ca																	1.4	2.4	1.6	4.6
Sr					33.3	40.8														
Zr	19.6	20.1															1	0.5	1.5	2
Ba									27.9	18.8	17	14.7	18	17.1	20.1	21.9	16.3	17.5	13.2	14.3
Yb			13.2	11.8													11.7	10.9	8.9	9.4

The compositional changes after incineration are minor compared with the differences between groups. The elemental concentrations listed are an average of four separate area analyses at × 500.

retrieval, analysis, and dental record comparison. Record comparison reemphasizes the need for practitioners to be diligent in their record keeping in patient charts and include all relevant information with respect to treatment including the type (brand name) of restorative material used.

It is recognized that this study is limited to a subset of the resins currently on the U.S. market. As such, this work represents the initial stages of database generation. However, the importance of this work lies in demonstration of the diversity of materials used today that could be unique identifiers. Knowledge of this can be useful in forensic investigations (5).

The analysis was performed using standard equipment and conditions, and comparable results should be obtainable in laboratories worldwide.

These findings, therefore, are of significance to the forensic community. Further work in this area will expand the database of resins and will include other restorative materials.

Addendum

The South Campus Instrument Center, University at Buffalo, School of Dental Medicine, is available to perform analyses of the nature described in this article. Submissions should be directed to the corresponding author.

Acknowledgments

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