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TECHNICAL NOTE

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Identification of Incinerated Root Canal Filling Materials After Exposure to High Heat Incineration

ABSTRACT: With the increase in global terrorism there is a higher probability of having to identify victims of incineration events secondary to incendiary explosive devices. The victims of incineration events challenge forensic odontologists when coronal restorations are no longer present to compile postmortem data. With 40 million root canals being completed annually in the United States, a very large pool of antemortem data is available to the forensic odontologist to make positive identifications. When complete and thorough dental records exist, individuals that have undergone surgical and nonsurgical root canal therapy may have materials present in the canal that may aid in identification. This study provides elemental fingerprints of root canal obturation materials to be utilized as a forensic identification aid. This study used scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) to assess the elemental composition of materials before and after high temperature incineration. Sixteen endodontic materials were analyzed pre-incineration and placed in extracted teeth. The filled teeth were subjected to incineration at 900°C for 30 min to simulate incineration events or cremation. Incinerated materials were radiographed and re-analyzed to determine if they retained their original elemental composition. Endodontic sealers, gutta percha, root-end filling materials, silver points, and separated files were distinguishable in the canal and traceable after incineration. The authors present a fingerprint of the endodontic obturation materials that are capable of withstanding high heat incineration to be used as an aid for postmortem identification. This work represents the initial stage of database generation for root canal filling materials for use as an aid in forensic identification.

KEYWORDS: forensic science, forensic odontology, endodontic materials, victim identification, SEM/EDS

Identification of victims of incineration events is a daunting and intensive task that requires the coordination of professionals from a number of disciplines. Victims of incineration events result from airline accidents, automobile accidents, bombings, or wrongful cremation. In the United States, there are over 43,000 highway fatalities annually, 4500 fire deaths per year, and *c.* 14,300 worldwide terrorist attacks resulting in over 20,400 deaths (1–3). Airline accidents and terrorist bombings involve many victims leading to a mass casualty situation where the identification of victims proves to be an overwhelming task using traditional forensics. Identification of victims of incineration events or wrongful cremation compounds this dilemma for the forensic odontologist trying to establish a positive postmortem identification. The central dogma of forensic dental identification is comparative analysis of antemortem dental records with postmortem dental remains.

The American Academy of Forensic Sciences recognizes forensic odontology as a specialty concerned with bite-mark identification in criminal cases and the identification of humans using antemortem dental records (4). The identification of humans utilizes the theory that all individuals are unique and can be identified using the anatomical differences of their dentition or the iatrogenic modification made by oral healthcare providers.

Historically, positive identification of unidentifiable incinerated passengers was made after a plane crash in the Taieri Gorge of New Zealand. The two corpses were positively identified using a full mouth series of radiographs to identify previous composite and amalgam restorations as guidelines (5). Similarly, 36 victims were positively identified after a Dash 7 airline misjudged altitude and crashed into a mountain in Northern Norway. The victims were completely incinerated due to the impact and exposure to high temperatures. Many of their teeth lost their enamel and amalgam restorations melted. Supraosseous preparations in dentin were compared to antemortem dental records to make positive identifications of the victims (6). Mass casualty events arise when hundreds of incinerated victims need to be identified. This occurred in 1982 when Pan Am flight 759 crashed and only 75% of the 153 victims were positively identified using dental remains (7).

Incineration events can have temperatures as high as 1100°C and standard cremation procedures routinely employ temperatures from 870–980°C for 1–1.5 h (8,9). It has been documented that enamel, dentin, and cementum all exhibit different behaviors dependent on the intensity and exposure time to the heat. Muller et al. (10) describe the total separation of the enamel shell at temperatures of 450°C due to a lower rate of shrinkage secondary to a lower water content when compared to dentin. Similar findings by Merlati et al. (11) indicated that complete separation of the coronal dentin and enamel occurs at 1100°C but roots remain intact and covered with cementum. Restorative and esthetic dental materials found in many coronal restorations used to identify nonincineration victims may be unreliable for identification of incineration victims due to the excessive distortion due to shrinkage and their propensity to fall out (12). Bush et al. (13) show similar findings of lost coronal restoration in their study of composite restorations in cremains. These studies indicate that high temperature incineration events make the

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coronal restoration an unreliable means for positive victim identification. Osseous periradicular tissues and the soft tissues of the oral cavity protect root structure and endodontic materials placed therein making it possible for the forensic team to identify the individual by the treated root canal.

Forty million root canal treatments are performed annually in the United States and 97% of endodontically treated teeth remain in the mouth after 8 years (14). It is widely accepted that endodontic therapy involves the thorough cleaning and shaping of the root canal system followed by the three-dimensional obturation of the root canal system. Various techniques and materials are employed depending on the clinician's training or practice philosophies, but the result is a radiopaque obturation material that is placed as an orthograde or retrograde filling.

Nonsurgical endodontic therapy is completed with the placement of an orthograde obturation material that possesses radiopacity, biocompatibility, retrievability, insolubility, and thermoplasticity. Gutta percha is the most commonly used core material and consists of formulations of zinc oxide, gutta percha, waxes, and heavy metal radiopacifiers (15). Manufacturers of gutta percha vary formulations only slightly, thus making most all gutta percha brands similar in composition. Gutta percha was unsuccessfully challenged by Jasper (16) who started obturating with silver points in an attempt to better control the apical extent of the material within the canal. Recently, resin obturation materials are used and utilize a resin sealer to create a "monoblock" of solid material within the root canal system. These products are similar in composition and are available as Epiphany (Pentron Clinical Technologies, Wallingford, CT), InnoEndo (Heraeus-Kulzer, Armonk, NY), Resinate (Obtura Spartan, Fenton, MO), SimplyFill (Lightspeed Technology, Inc., San Antonio, TX), and RealSeal (Sybron Endo, Orange, CA).

There is a multitude of endodontic sealers that fill the space between the radicular dentinal wall and the core material. Commonly used sealers are zinc oxide-eugenol formulations, calcium hydroxide, glass ionomers, and resin sealers. Kerr EWT (Kerr Corporation, Orange, CA) is a zinc oxide-eugenol-based sealer and is used for its antimicrobial activity, tackiness, and long working times. Tubliseal (Sybron Endo) is also a zinc oxide-eugenol-based sealer that sets faster due to the addition of a catalyst. Most sealers include silver particles, barium sulfate, and/or bismuth to increase their radiopacity. Apexit (Ivoclar Vivadent, Amherst, NY) is a zinc oxide-eugenol sealer with calcium hydroxide added to take advantage of possible antimicrobial properties and to facilitate cementogenesis and osteogenesis at the apical foramen.

During the course of endodontic treatment untoward events occur when endodontic instruments separate in the canal. Many of these separated instruments are unrecoverable and become incorporated into the root canal filling material. Natkin and Crump (17) reported that a separated instrument does not significantly decrease the success rate of the root canal depending on when and where the separation occurred. Endodontic files are primarily made of stainless steel or nickel titanium, each imparting a different unique characteristic to the procedure. Incorporation of the instrument into the obturation would create a unique elemental fingerprint and radiographic image.

When nonsurgical endodontic therapy fails, it is most likely due to instrument separation, blocked canals, ledges, or infected canals. The clinician considers surgical endodontic therapy where the root end is resected and a retrofilling is placed to seal the root canal system. Retrofilling materials should be dimensionally stable, radiopaque, and insoluble, while also being well tolerated in the periradicular tissues. Commonly placed root-end filling materials are Grey or White ProRoot MTA (Dentsply, Tulsa, OK) and Super-EBA (Harry J. Bosworth Co., Skokie, IL) (18,19).

The aim of this study was to utilize scanning electron microscopy/energy dispersive X-ray spectroscopy (SEM/EDS) to assess the identification and composition of endodontic obturation materials in human teeth exposed to high heat incineration. The results of this study can be used as an aid in forensic identification.

Materials and Methods

Eight endodontic sealers and three root-end filling materials were mixed according to the manufacturer's instructions and allowed to set for 3 weeks. Two gutta percha cones and a silver point were also used in this study. Table 1 lists all the obturation materials used in this study and the compositions of the sealers according to the manufacturer or the material safety data sheet (MSDS). Each material was analyzed by SEM/EDS to determine their unique composition as a baseline prior to obturation and incineration.

The next part of this investigation utilized 16 single-rooted extracted teeth. The teeth were disinfected in 5.6% sodium hypochlorite for 1 h and stored in sterile water at room temperature. The teeth were systematically accessed with a #4 carbide round bur in a high speed handpiece with water irrigation. The teeth were initially instrumented with a #10 Flex-R (Miltex, York, PA) stainless steel hand file to establish a working length by subtracting 1 mm from where the file protruded from the apical foramen. All canals were cleaned and shaped using ProTaper (Dentsply) rotary files up to the F3. All teeth were irrigated with 2.6% sodium hypochlorite and ethylene diaminotetraacetic acid (EDTA) to remove the smear layer and dried prior to obturating the teeth.

Ten teeth were obturated with Dentsply medium gutta percha cones and sealers listed in Table 1 using a single cone technique to insure homogeneity of the gutta percha. Three teeth were completely obturated with the root-end filling material, one tooth was obturated with Epiphany sealer and a medium Epiphany point, one tooth was obturated with a silver point and Kerr EWT sealer. Lastly, one tooth had a size 20 Profile nickel titanium file separated midroot while another had a size 20 Flex R stainless steel file separated midroot.

After obturation, the teeth were radiographed using a digital sensor, then remained in 100% humidity for 3 weeks to allow the sealers to set. The filled teeth were set in a ceramic crucible in a burnout oven and heated at 900°C in air for 30 min. After removal from the burnout oven the teeth were carefully radiographed digitally and viewed under the stereomicroscope to determine if the obturation materials were distinguishable.

The pre-incinerated samples and the postincinerated teeth were analyzed using a Hitachi S-4000 Field Emission SEM equipped with an IXRF 500 X-ray microanalysis system. EDS spectra were

TABLE 1—*Brands of obturation material and composition as provided by the manufacturer.*

Obturation Material	Elemental Composition
AH 26 (Dentsply)	Ag, Bi, Ti
AH Plus (Dentsply)	Zr
Apexit (Ivoclar)	Ca, Zn
Epiphany sealer (Pentron)	S
Epiphany point (Pentron)	Ba, Bi, S
EZ Fill (EDS, Hackensack, NJ)	Undetermined
Gutta Percha (Dentsply)	Zn
Gutta Percha (Sybron Endo)	Ba, S, Zn
Kerr EWT (Kerr)	Ag, Zn
Nogenol (GC America, Alsip, IL)	Ba, Bi, S, Zn
ProRoot White MTA (Dentsply)	Undetermined
ProRoot Grey MTA (Dentsply)	Al, Bi, Ca, S, Si
Super EBA (Harry J. Bosworth Co.)	Undetermined
Tubliseal (Sybron Endo)	Ba, S, Zn

collected from area scans at 1000–2500× magnification. The variation in magnification was used to restrict analysis to the material in question. Conditions of analysis were 25 keV acceleration voltage at a 43° takeoff angle, for 100 s live time. Dead time was typically 5–7% at count rates of 1000–1500 cps. The detection limit for most elements using this instrumentation is around 1%. The EDS spectrum was obtained and used qualitatively to determine which elements could be detected in the sample above the detection limit. Thus, only the presence of elements in the major and minor concentrations is reported. Elemental concentrations were not quantified, although the data collected could be analyzed by semi-quantitative software. SEM images were analyzed to observe and annotate any distinguishing characteristics of the materials.

Results

Pre-incineration

The elemental analysis of gutta percha, sealer samples, root-end filling materials, silver point, and the separated files all exhibit unique and identifiable elemental characteristics within this class of materials. Table 2 illustrates the initial pre-incineration elemental composition determined by the SEM/EDS analysis. Pre-incineration radiographs of all teeth show complete obturation of the single rooted teeth with intact enamel, dentin, and cementum. Characteristically straight and serrated radiopaque lines clearly indicate represent the separated files.

Postincineration

Similar to Merlati et al.'s (11) study, all incinerated teeth changed morphologically as the enamel and coronal dentin quickly dehydrated and splintered off the tooth structure. The residual root structure was chalky in appearance and extremely brittle. Figure 1 illustrates radiographically how the obturated teeth lost their coronal structure due to the rapid dehydration and shrinkage of the dentinal tubules. The radiographs also illustrate how the teeth maintained their radicular structure and the obturation materials contained within. Stereomicroscopic analysis indicated that the obturation materials were distinguishable from root dentin and elemental analysis could be conducted. Figure 2 illustrates the clearly distinct regions of the radicular dentin, Apexit sealer, and Dentsply gutta percha.

TABLE 2—*Pre-incineration SEM/EDS elemental analysis.*

Obturation Material	Elemental Analysis
AH 26 (Dentsply)	Ag, Bi, Ti
AH Plus (Dentsply)	Ca, W
Apexit (Ivoclar)	Bi, Ca, Si, Zn
Epiphany sealer (Pentron)	Ca, Ba, Bi, S, Si
Epiphany point (Pentron)	Ba, Bi, Ca, Si
EZ Fill (EDS)	Bi
Gutta Percha (Dentsply)	Zn
Gutta Percha (Sybron Endo)	Ba, Si, S, Zn
Kerr EWT (Kerr)	Ag, Zn
Nogenol (GC America Inc.)	Ba, S, Zn
Profile NiTi File– 25 (Dentsply)	Ni, Ti
ProRoot Grey MTA (Dentsply)	Al, Bi, Ca, Fe, Si
ProRoot White MTA (Dentsply)	Bi, Ca, Si
Silver point	Ag
Stainless steel file– 20 (Miltex)	Cr, Fe, Ni
Super EBA (Harry J. Bosworth Co.)	Al, Zn
Tubliseal (SybronEndo)	Ba, S, Si, Zn

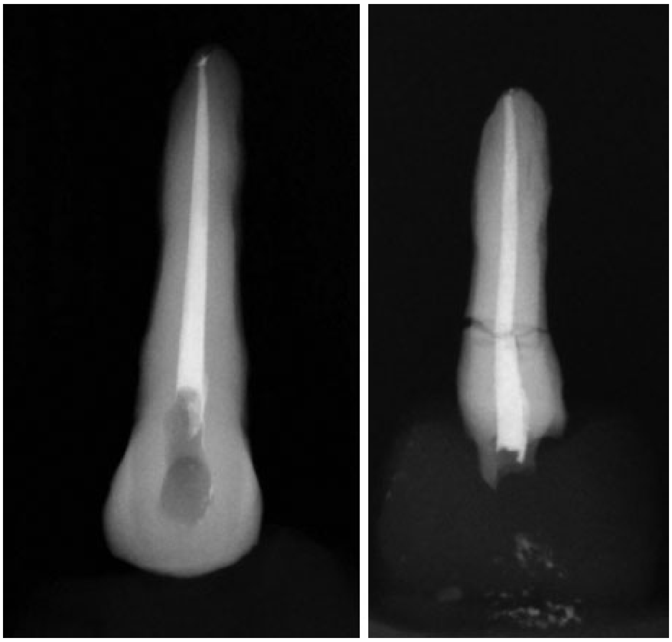

FIG. 1—*Pre-incineration radiograph of AH Plus sealer (left). Postincineration radiograph of AH Plus sealer (right).*

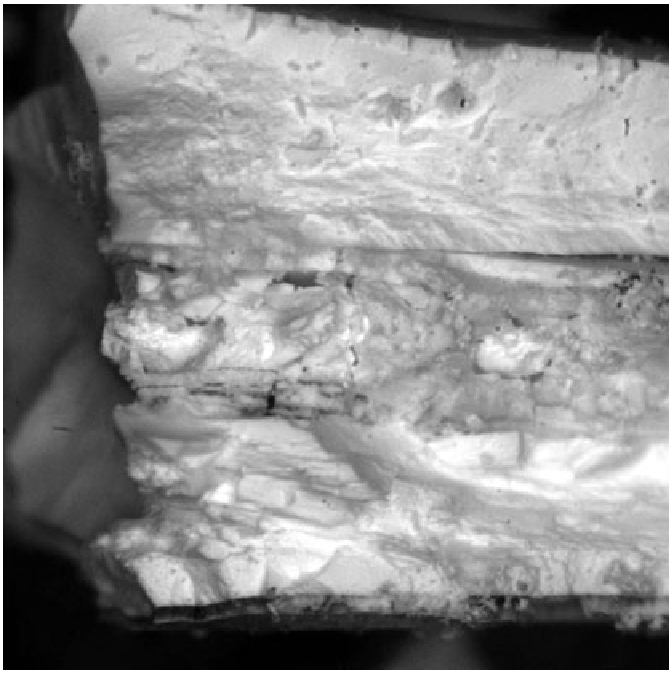

FIG. 2—*Stereomicroscope view of Apexit sealer, Dentsply gutta percha, and dentin in an incinerated root.*

Careful manipulation of the brittle root structures allowed SEM/EDS analysis of the root filling materials. Similar to the stereomicroscope, images created by the SEM illustrate the distinct regions of sealer, gutta percha, and radicular dentin as noted in Fig. 3. Likewise, the silver point, stainless steel file, and nickel titanium file can be readily identified for further elemental analysis (Figs. 4–6). A titanium oxide film formed on the surface of the nickel titanium file as seen in Fig. 6. Distinctions were sometimes difficult to make between the sealer and the gutta percha. The

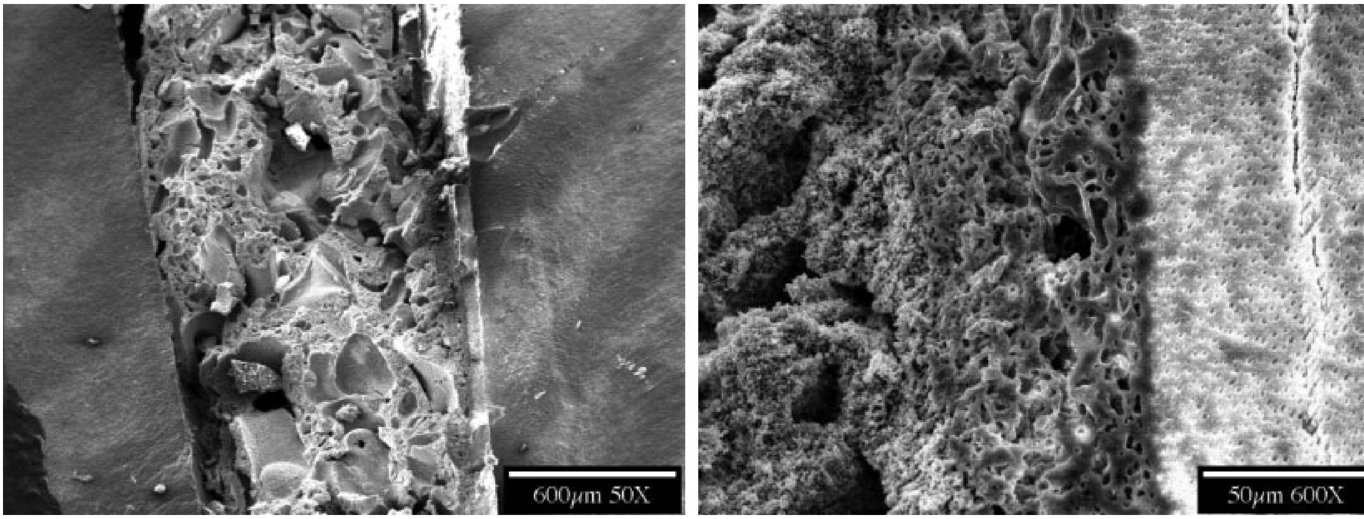

FIG. 3—*Scanning electron microscopy images of Apexit sealer used with Dentsply gutta percha. Both magnifications show clear distinction between the gutta percha* (*left*), *sealer* (*middle*), *and dentin* (*right*) *after incineration.*

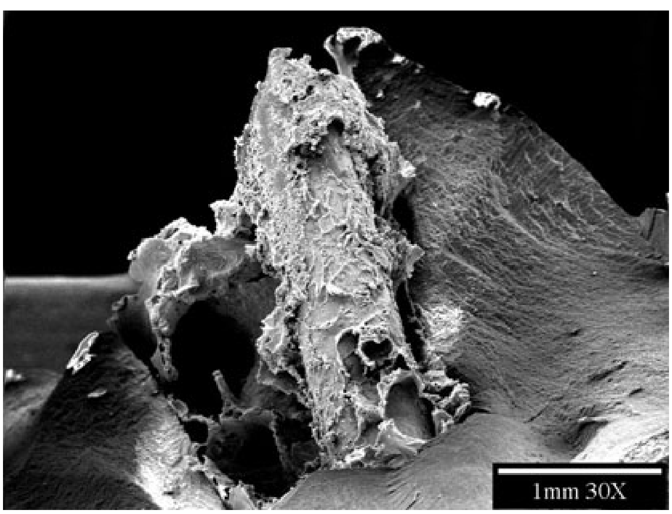

FIG. 4—*Scanning electron microscopy image of a silver point and Kerr EWT sealer protruding from the fractured incinerated tooth.*

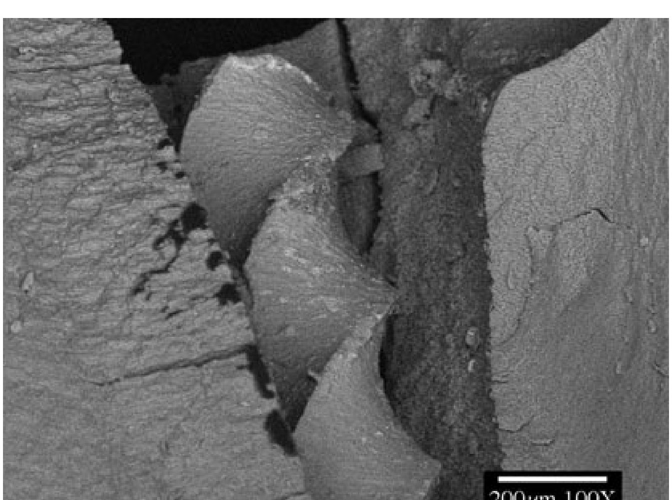

FIG. 5—*Scanning electron microscopy image of #20 stainless steel file placed in the canal.*

Epiphany obturation method created a monoblock mass of obturation material and sealer and was analyzed as one mass (Fig. 7). Grey ProRoot MTA was detectable in the root canal system as illustrated in Fig. 8 and is unique in composition when compared to white ProRoot MTA.

Table 3 lists the elemental composition of different sealers after incineration. Table 4 lists elemental composition of the common root-end filling materials, the silver point, and the separated files after incineration. It is of interest to note that the postincineration elemental compositions of all root canal filling materials contain phosphorous and calcium due to inorganic composition of radicular dentin.

Discussion

This study demonstrates that complete, thorough, and accurate antemortem dental records could be used to identify individuals that have undergone surgical or nonsurgical root canal therapy based upon elemental analysis of the obturating material. Elemental fingerprints of the specific endodontic file, sealers, or cement can prove to be an essential tool for the forensic odontologist.

The increase in the number of clinicians using nickel titanium files to clean and shape the root canal will inevitably lead to more instrument breakages in the canal. Nickel titanium is a superelastic material that retains shape memory to negotiate canals, bends, or root dilacerations. This material was invented by the Naval Ordinance Laboratories under the name of Nitinol and was later utilized in dentistry for orthodontics and in endodontic files by Walia et al. (20). A severe disadvantage of this material is that it is limited by its cyclic and torsional fatigue properties. A recent retrospective study reported a 1.68% file separation rate in a postgraduate endodontic residency program and earlier studies report separation rate to be as high as 6% (21). It can be assumed that the failure amongst general practitioners may be even higher. The radiographic image and SEM/EDS analysis clearly identifies nickel titanium files after incineration due to its straight radiographic appearance in canal curvatures and the elemental fingerprint of titanium.

The SEM identified a film coating the surface of the nickel titanium file after exposure to high heat within the root canal system. Figure 6 illustrates a rough, "glacier-like" surface coating on the nickel titanium file under high magnification. This film was formed

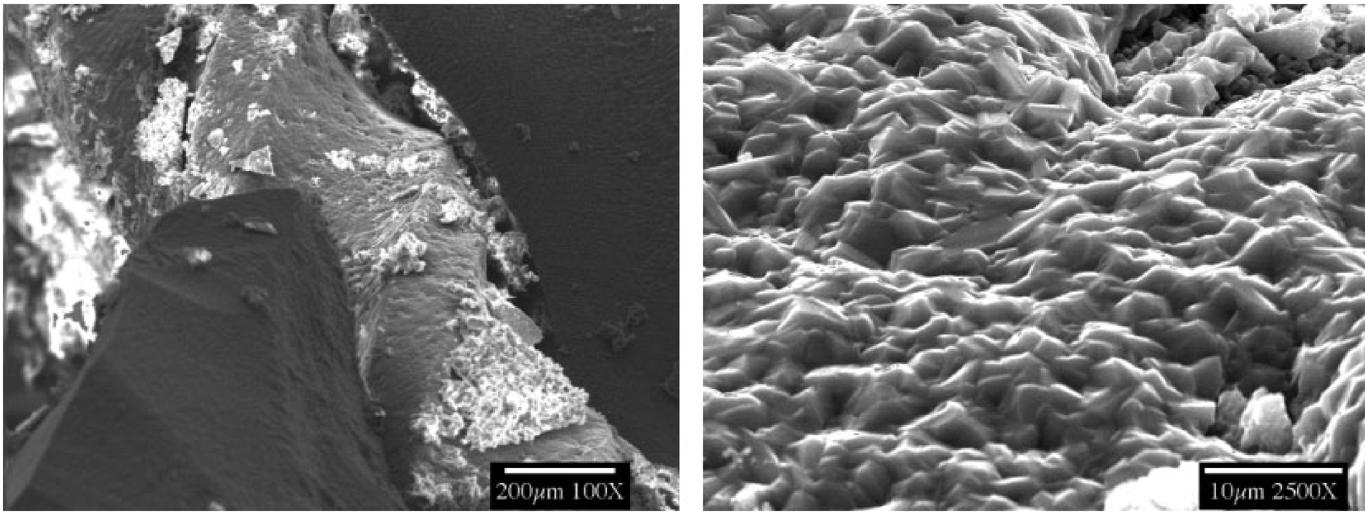

FIG. 6—*Scanning electron microscopy image of a separated Profile nickel titanium rotary file in incinerated root canal. The higher magnification image at right shows detail of the surface oxide film.*

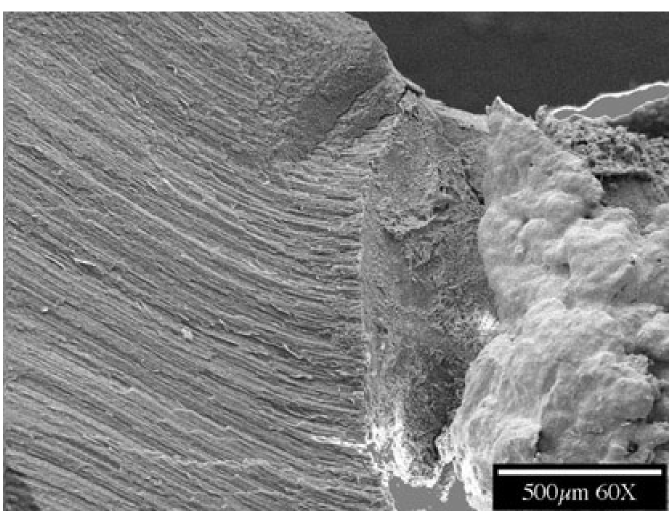

FIG. 7—*Scanning electron microscopy image of Epiphany resin obturation material maintaining one complete block of sealer and obturating material after incineration.*

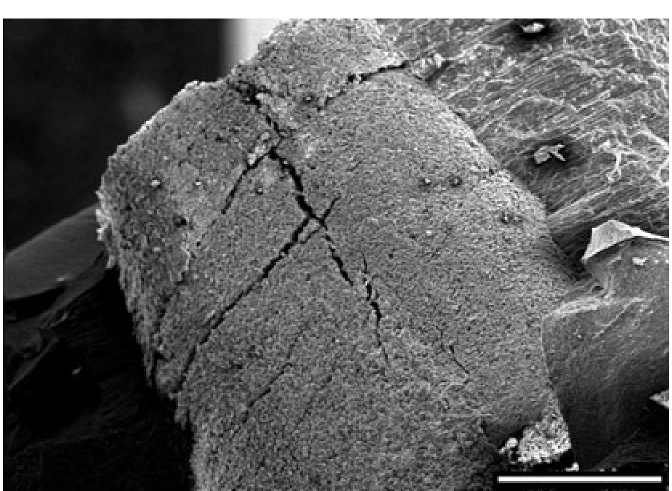

FIG. 8—*Scanning electron microscopy image of grey ProRoot MTA after incineration.*

TABLE 3—*Postincineration sealer and gutta percha SEM/EDS analysis.*

Obturation Material	Elemental Analysis
AH 26 (Dentsply)	Bi, Ca, P, Ti, Zn
AH Plus (Dentsply)	Ca, P, W
Apexit (Ivoclar)	Ca, Si, Zn
Epiphany Monoblock (Pentron)	Ba, Bi, Ca, Ca, P, Si
EZ Fill (EDS)	Bi, Ca, P
Gutta Percha (Dentsply)	Zn
Gutta Percha (Sybron Endo)	Ba, Si, S, Zn
Kerr EWT (Kerr)	Ag, Zn
Nogenol (GC America Inc.)	Ba, Ca, P, S, Zn
Tubliseal (Sybron Endo)	Ba, Ca, P, S, Si, Zn

TABLE 4—*Postincineration root-end filling material, silver point, and separated file SEM/EDS analysis.*

Obturation Material	Elemental Analysis
Profile NiTi File– 25 (Dentsply)	Ca, P, Ti
ProRoot Grey MTA (Dentsply)	Al, Bi, Ca, Fe, P, Si
ProRoot White MTA (Dentsply)	Bi, Ca, P, Si
Silver point	Ag, Zn
Stainless steel file– 20 (Miltex)	Ca, Cr, Fe, Ni
Super EBA (Harry J. Bosworth Co.)	Al, Ca, P, Zn

by the separation of titanium from the alloy at high temperatures followed by a preferential oxidation at the surface.

The nickel titanium instrument separation rate is seven times higher than stainless steel files (21). Stainless steel files are commonly used in endodontic treatment to negotiate the canals, create a "glide path" for the rotary nickel titanium instruments, and to clean and shape the canal system. The separated hand files will commonly be smaller and found apically in areas of greater canal constriction, but due to their chromium, iron, and nickel content, they can be positively fingerprinted using SEM/EDS.

The sealers analyzed were fingerprinted by their unique elemental constituents and specific heavy metal content. Heavy metals are used in endodontic obturation materials to impart radiopacity. Elements with higher atomic number and large nuclei will absorb X-rays more than metals with smaller nuclei. Grey ProRoot MTA, AH26, Apexit, Epiphany, and EZFill all contain detectable amounts of bismuth whose atomic weight is 208, thus making it more radiopaque than tooth structure. Other obturating materials such as Epiphany, Sybron Gutta Percha, Nogenol, and Tubliseal contain the heavy metal barium. Barium, whose atomic weight is 138 is also ideally suited for absorbing X-rays. Tungsten, whose atomic weight of 183 is unique to AH Plus sealer, is used for its radiopaque properties and as a catalyst to facilitate the autocure reaction of the epoxy sealer. Other identifiable elements include silver, which is found in Kerr EWT, Silver points, and AH26. Silver is used as a radiopacifier in the root obturation material. Aluminum is found in grey ProRoot MTA and Super EBA, whereas silicon can also be found in many different sealers, gutta percha, and root-end filling materials.

Advancements in microsurgical techniques, illumination, and biocompatible materials have given the practitioner the ability to perform root-end fillings and repair root perforation with success rates ranging from 91.5% to 96.8% (22). Mineral trioxide aggregate (MTA) has gained popularity as a root-end filling and perforation repair material due to its biocompatibility and its apparent cementoconductive characteristics (23). Grey ProRoot MTA has a different fingerprint from white ProRoot MTA due to differences in the concentrations of aluminum oxide and iron oxide, as expected from previous studies conducted by Asgary et al. (24). The various oxides will enable the forensic odontologist to properly ascertain if white or grey MTA was used based upon its elemental composition, radiographic appearance, and its placement in the root.

During high heat accidents or blast injuries, it is possible for temperatures to rise well above 900°C in relatively fast periods of time. Intense flash fires cause the pulp to boil and eventually explode the crowns of teeth leaving the tooth broken off at the gingival margin without any coronal restoration to help identify the victim (25). This would however leave root anatomy unaltered, and in the case of root canal therapy, the canal contents exposed and accessible for elemental analysis (25). It is of importance to recognize that enamel and coronal dentin will shrink at various rates causing the crown of the tooth and any possible distinguishing restorations to fall out. The roots of teeth are protected by the insulating properties of the maxilla or the mandible, the gingiva, the muscles of facial expression or mastication, and the skin of the face.

As roots are unique in their anatomy and obturating material, it is imperative that the forensic odontologist be aware of the unique anatomy including dilacerations or additional roots. In severe cases where teeth have avulsed from the maxilla or mandible, a replica of the shape of the root can be made by filling the empty socket with dental alginate mixed with barium sulfate prior to taking the radiograph (25). This will allow for better visualization of unique or identifying root anatomy.

It is a premise of this study that antemortem dental records list the type of dental obturating material; however, this type of meticulous record keeping is not always routinely practiced. The responsibility of the dentist is not only to the immediate oral health care of the patient but also the unfortunate event where a patient may need to be identified based on their antemortem records. Therefore, it is strongly encouraged that clinicians include brand name of any dental materials placed in the patients.

This investigation examined many of the root canal filling materials available on the market that can be expected in the treated root canal system. It may be practical to include composite resins in the differential analysis of root canal filling materials as these materials can be inadvertently placed into the root canal space and may contain similar heavy metals like barium to aid in their radiopacity. Bush et al. (26) determined that effective elemental analysis would isolate silicon, strontium, ytterbium, and other unique elements that could differentiate composite resins.

This study did not take into consideration variations in lots of the same brand of material. It would be prudent for future research to test multiple lots by a quantative analysis to determine if the consistency varies. Bush et al. (13) positively identified dental restorative materials using the portable X-Ray Fluorescense spectrometer (XRF). Studies using the portable XRF to positively fingerprint endodontic materials would be advantageous to the forensic team as it would eliminate the large, expensive laboratory-based SEM/EDS to conduct elemental analysis of postmortem remains. XRF spectroscopy would ideally be suited for investigations in a contaminated or dangerous field setting.

This database of the pre- and postincinerated elemental analysis of endodontic filling materials can be kept as an immediate reference for the forensic odontologist. It is an initial starting point and can be completed with other materials including pastes, glass ionomers sealers, gates-glidden burs, lentulo spirals, and many different types of posts that are found in canals of teeth.

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