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Rapid and non-destructive analysis of metallic dental restorations using X-ray fluorescence spectra and light-element sampling tools

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ABSTRACT

Introduction: Recently, allergic diseases caused by dental metals have been increasing. Therefore, rapid and accurate analytical methods for the metal restorations in the oral cavities of patients are required. The purpose of this study was to develop a non-destructive extraction method for dental alloys, along with a subsequent, rapid and accurate elemental analysis.
Materials and method: Samples were obtained by polishing the surfaces of metal restorations using a dental rotating tool with disposable buffs and polishing pastes. As materials for the analysis, three dental alloys were used. To compare the sampling and analysis efficiencies, two buffs and seven pastes were used. After polishing the surface of a metal restoration, the buff was analyzed using X-ray scanning analytical microscopy (XSAM).
Results: The efficiency of the analysis was judged based on the sampling rate achieved and the absence of disturbing elements in the background in fluorescence X-ray spectra. The best results were obtained for the combination of TexMet as a buff with diamond as a paste. This combination produced a good collection efficiency and a plain background in the fluorescence X-ray spectra, resulting in a high precision of the analysis.

1. Introduction

In dentistry, more than 20 metallic elements are processed into various dental metal alloys. These alloys are then cast and processed for use as metal restorations.

Fleischmann [1] first reported stomatitis from an amalgam restoration in the oral cavity in 1928. Various allergic diseases caused by intraoral metal restorations have since been reported [2–9]. Recently, an increase in allergic diseases has been reported in Japan, and metal allergies have attracted attention in dentistry [10]. The removal of metal restorations in the oral cavities of patients has become widespread in Japan, and an improvement in allergic diseases has been reported [11].

Patients with allergic diseases must be examined to determine whether a metal restoration contains the allergenic substance. The sampling method should be a non-destructive extraction from the metal restorations in the oral cavity along with a subsequent rapid and accurate elemental analysis.

The conventional extraction method has been to scrape powder from a metal restoration using a carbide bur. This powder is then collected on the tip of a fresh cotton swab and analyzed using an X-ray fluorescence spectrometer. However, this method not only damaged the structure of the metal restoration but also exacerbated the allergic reaction by scattering metal fragments [10].

Uo and Watari [12] used fresh silicone points (type M, Shofu) to sample dental metal, after which the metal-attached silicone points were analyzed using X-ray scanning analytical microscopy (XSAM) (XGT-2000V, Horiba). The amount of attached metal was estimated to be less than 30 μg. Although this method did not damage metal restorations, it was observed that Si and Ti were found because of the silicone point used. This is a disturbing factor for the X-ray fluorescence analysis.

The purpose of this study was to develop a non-destructive extraction method for dental metal restorations along with a subsequent rapid and accurate elemental analysis.

2. Materials and method

2.1. Sampling tool

Fig. 1a shows a schematic of a polisher disk with a disposable buff and polishing abrasive paste. After the disk was mounted on a rotating dental tool (contra-angle handpiece), as shown by the

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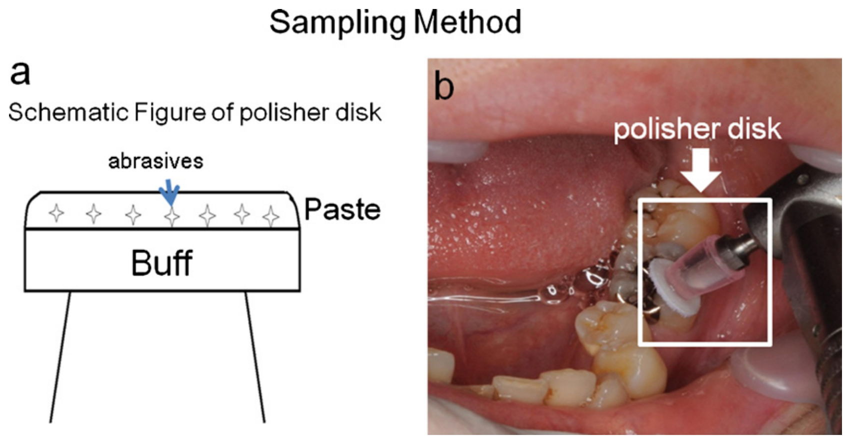

Fig. 1. Clinical sampling situation from dental prostheses set in oral cavity of patient. (a) A schematic of a polisher disk with a disposable buff and polishing abrasive paste. (b) Sampling method from patient.

arrow in Fig. 1b, samples were collected by polishing the surface of the metal restorations.

To compare the sampling and analysis efficiencies, two buffs and seven pastes were used.

2.1.1. Polishing buff disk

Two buffs (SuperSnap, Shofu) and (TexMet, Buehler) were used in this study. SuperSnap is composed of a synthetic velvet cloth, used for the final polish of dental prostheses of resin composites and porcelains. TexMet is non-woven pressed cloth, generally used to polish material object in industrials.

2.1.2. Polishing paste

Table 1 lists the various abrasives that the used pastes were composed of: (a) diamond (Diamond Paste, Scandia), (b) a mixture of diamond and titanium oxide (DirectDiaPaste, Shofu) used as received, (c, d) boron nitride (BN-B and BN-T, Showa Denko), and (e) alumina (Metapolish, Fujimi), as prepared in this study.

To make the pastes composed of powders of boron nitrides BN-B and BN-T, with Metapolish (α-Al2O3), as abrasives, Vaseline (white petrolatum) and glycerin were used as bonding solvents. BN-B and BN-T are highly crystalline cubic boron nitride super abrasives with a hardness close to that of diamond. The particle size distribution of BN-B was 0–1 μm (mostly about 1 μm from field emission scanning electron microscope observation as shown in the result) with an angular shape, while that of BN-T was 0–2 μm (mostly about 2 μm in a similar way) with a block-like shape. This data provided by supplier.

The solvent/powder volume ratio was set to 50/50.

Table 1
Various pastes used for sampling of metal restoration.

Paste name (supplier)	Abrasives	Abrasive particle size	Solvent
Diamond Paste (Scan-Dia)	Diamond	7 μm[a]	Oil
DirectDiaPaste (Shofu)	Diamond + TiO_2 (anatase)	Diamond 2–4 μm[a]	–
BN-B (Showa Denko)	BN	0–1 μm[a]	Vaseline/glycerin
BN-T (Showa Denko)	BN	0–2 μm[a]	Vaseline/glycerin
Metapolish (Fujimi)	α-Al2O3	2 μm[a]	Vaseline/glycerin

[a] Manufacturer's providing values.

2.1.3. Dental alloys for sampling

Three dental alloys were used as analysis materials: Au–Ag–Pd (Castwell, GC), Ni–Cr (Betalloy, Yada Kagaku Kougyo), and silver alloy (Milosiver, GC). The commercial names and compositions of these alloys were Castwell (12Au–46Ag–20Pd–20Cu–2(Zn, In, Ir)), Betalloy (62Ni–12Cr–15Cu–8Mo–3others), and Milosiver (65Ag–15Zn–20Sn). To ensure the same surface states for all of the dental alloys, the surfaces were polished using a silicone point set on a dental rotating tool before sampling.

2.2. Sampling method

Sampling was performed directly with each dental alloy by simulating the method used for a patient's oral cavity in a clinic. The sampling time was 30 s, and the imposed pressure was about 10 gf.

2.3. Observation and analysis

After polishing the surfaces of the metal restorations, the X-ray fluorescence spectra of the buffs were analyzed using XSAM. X-rays were generated from a rhodium (Rh) cathode irradiated by electrons at 50 kV and 1 mA. The spatial resolution of the analysis was 100 μmφ, which was equal to the X-ray beam size formed by the 100 μmφ X-ray guide tube (XGT) of the XSAM, and the measurement time was 120 s.

The collected amount of dental alloy was determined from weight difference of the buff before and after sampling. Randomly-extracted five samples were measured by Micro Balance (AEM-5200, Shimadzu).

The buffs and pastes were also analyzed, prior to their use, with the help of XSAM and X-ray diffraction (XRD) (Multi Flex, Rigaku) and observed by field emission scanning electron microscope (FE-SEM) (S-4000, Hitachi high technologies).

3. Results

3.1. Sampling method

The total time required for the entire analysis and sampling process was under 10 min. The collected amount of dental alloys was less than 30 μg.

3.2. Buff

Fig. 2 shows the SEM images of the buffs: (a) TexMet (TM) and (b) SuperSnap (SS). The TM buff was made from numerous fibers bound