COMPONENT ANALYSIS OF DENTAL PORCELAIN FOR ASSISTING DENTAL IDENTIFICATION

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ABSTRACT

The fluorescence of porcelain crowns recovered from the mouth of an unknown murder victim, and several control porcelain samples, were examined by fluorescent examination lamps. The fluorescence from two of the control samples was quite similar to that from the porcelain crowns recovered from the victim. To increase the objectivity of the results by quantitative analysis, the composition of each porcelain crown and control sample was also evaluated by wave dispersion X-ray microanalyser. The elements detected from the porcelain crowns of the victim matched those of two of the porcelain samples. Later, the antemortem dental records and radiographs of the victim were obtained through a dentist, who had recognized the name of the porcelain manufacturer in a postmortem dental information request placed on the Japanese Dental Association web page. Although component analysis of dental porcelain may be an effective means of assisting dental identification, a more rapid and non-destructive analysis for detecting the elements is required. The energy dispersive X-ray fluorescence (EDXRF) spectrometer was used for a pilot study of identification of porcelain composition.

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INTRODUCTION

The detection of tooth coloured restorations can be difficult under certain conditions in both controlled mortuary environments and mass fatality incidents managed outside of regular facilities.[1-3] White or composite filled teeth look similar to natural teeth under natural light, but under UV light they exhibit different features to those of natural teeth.[4] By using these characteristics, some fluorescent examination methods for identifying composite restorations have been discussed in the forensic dental field.[5-7] These characteristics are also available to identify various dental porcelain products. Both the detection of white dental materials and porcelain products in an unknown body by fluorescent examination may lead to a successful identification,[8,9] but unfortunately the evaluation of fluorescence must be carried out subjectively by the naked eye and is extremely difficult in a small sample. This may limit its application in some situations.

A previous case at our institution involved aesthetic dental porcelain veneer jacket crowns and porcelain fused to metal crowns found in the mouth of a skeletonised, unknown, murder victim.[10] Initially no antemortem dental information could be located. It was suggested to police that information about the victim might be obtained through dental technicians and porcelain manufacturers. As a screening, fluorescence examination was performed, comparing the recovered dental porcelains against several collected porcelain samples. Fluorescence from two brands of porcelain samples was quite similar to that from the porcelain veneer jacket crowns found in the victim.

To establish objective results by which to identify the products, the composition elements of each of the porcelain crowns were also evaluated by wave dispersive X-ray microanalyser (WDX). The elements comprising the samples from the victim matched those of two of the reference porcelain samples. We believe, as a result of this experience, component analysis of dental porcelain may be an effective means of assisting dental identification.

Using a wave dispersive X-ray microanalyser (WDX) for detection of elements involves much time-consuming sample preparation, requiring the sample porcelain to be cross-sectioned, polished to a smooth surface, and then covered with a thin film of carbon to avoid charging of the insulating targets.[11] This means that the dental evidence is destroyed and this technique is, therefore, not necessarily the best method to identify dental products. In addition, no database of constituent elements of dental porcelain products has been developed for forensic use. As an alternate investigative technique an energy dispersive fluorescent X-ray (EDX) spectrometer was used for fluorescent X-ray analysis. This instrument can perform quick and precise non-destructive qualitative and quantitative analyses of elements.

The aims of this study were to establish preliminary composition data of dental porcelains suitable for applying in dental identification, and to develop a convenient and non-destructive method of fluorescent X-ray analysis.

MATERIALS AND METHODS

Sample collection and preparation

Porcelain fused to metal restorations are the most frequently used restoration in Japan for the complex restoration of anterior teeth. From over 10 manufacturers of dental porcelain for the production of porcelain-fused-to-metal restorations in Japan, one was selected to provide samples for this study.

In building up the crown form to satisfy esthetic requirements, several kinds of porcelain powders must be used according to manufacturer's recommended procedures.[12] By accumulating thin layers of adequate enamel and dentin porcelains, appropriate transparency and opaqueness can be achieved. For the test analysis, the dental porcelains collected were the powders used around the crown surface (i.e. enamel and translucent porcelain powders). For the component analysis 14 kinds of porcelain powders (3 for enamel, 11 for translucency) were collected, as shown in Table 1. A total of 5 discs of each kind of porcelain powder were heat treated in a porcelain furnace (Sella-Fusion; Sekisui Co. Ltd.) using the manufacturer's recommended firing cycles. A total of 70 disc specimens were produced for this analysis.

Energy dispersive X-ray fluorescence

The energy dispersive fluorescent X-ray spectrometer [Rany EDX-900, (hereafter abbreviated as EDX900); Shimadzu Co., Japan] was used for the component analysis of the dental porcelains (Fig.1). The desktop type fluorescence X-ray spectrometer EDX900 consists of an analyzer, workstation, and printer. The analyzer irradiates X-rays onto a sample and detects generated fluorescence X-rays. The workstation processes the data by operating the analyzer. Quick and precise non-destructive qualitative and quantitative analyses of elements of a wide range from sodium ($_{11}$Na) to uranium ($_{92}$U) can be performed.

Component analysis of porcelain samples

Qualitative and quantitative analyses were performed at the centre of each of the 70 disc samples after confirming the setting position via a built-in CCD camera. The sampling depth of the method is approximately 1.0mm.

In the qualitative analysis, the specified elements were detected on the profile and their names displayed on the monitor automatically. Depending on measuring conditions, two or more fluorescent X-ray pulses may enter the sample at the same time. These cannot be separated to individual pulses and are signal-processed as a single pulse. As a result, a peak appears at a position higher than the actual energy position. To check for overlap of summed peaks and the peaks of other elements included in the sample, the energy table of individual elements was referenced against a final register of element composition.

Table 1: *Porcelain powders used for component analysis*

Porcelain Powder (type)	Batch Number
E_1 (Enamel)	OCX 19
E_2 (Enamel)	OCY 14
E_3 (Enamel)	OD 120
T_0 (Translucent)	OC 604
T_1 (Translucent)	OCY 30
T_2 (Translucent)	604 17
Tx (Translucent)	OB 706
LT_0 (Translucent	OCY 08
LT_1 (Translucent)	OCY 22
T•B (Translucent)	OCZ 04
S•B (Translucent)	OC 130
C•E (Translucent)	OC 314
C•W (Translucent)	806 17
I•A (Translucent)	806 17

Fig.1: *The energy dispersive fluorescent X-ray spectrometer (Rany EDX-900; Shimadzu Co., Japan)*

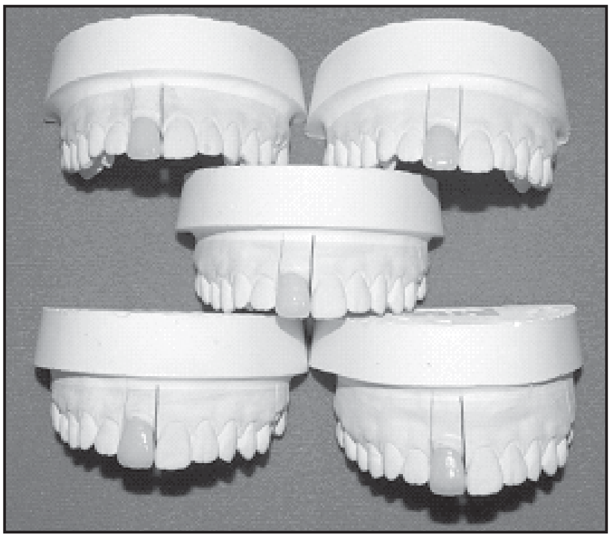

Fig.2: *Porcelain fused to metal crowns by 5 dental technicians*

In the Qualitative-Quantitative Analysis mode, quantitative calculation of an element detected by the qualitative analysis was carried out by the Fundamental Parameter method without using standard samples.

The main component of porcelain is feldspar and its chemical compositions were assumed to be some metallic elements combined with oxygen to form stable oxides. We, therefore, set the compound form as an Oxide with the following analytical conditions:

- Collimator size/ 1.0 mm
- Atmosphere/vacuum
- Voltage/ 15 ~ 50kV
- Current/ 100 µ A
- Integration Time/ 300 sec

The lower limit of detection by EDX900 in the qualitative-quantitative analysis mode was different in each element detected, as follows; Si: 500ppm, Ca:50ppm, Fe-U:10ppm.

The qualitative-quantitative analysis was carried out three times per disc sample on separate days of the week, and the mean value of measured wt% in each kinds of porcelain was calculated to determine element composition.

To study the reliability of this analysis, 5 dental technicians were asked to produce a porcelain-fused-to-metal crown on working casts using the same products in shade Vita-A3 (Fig.2). The

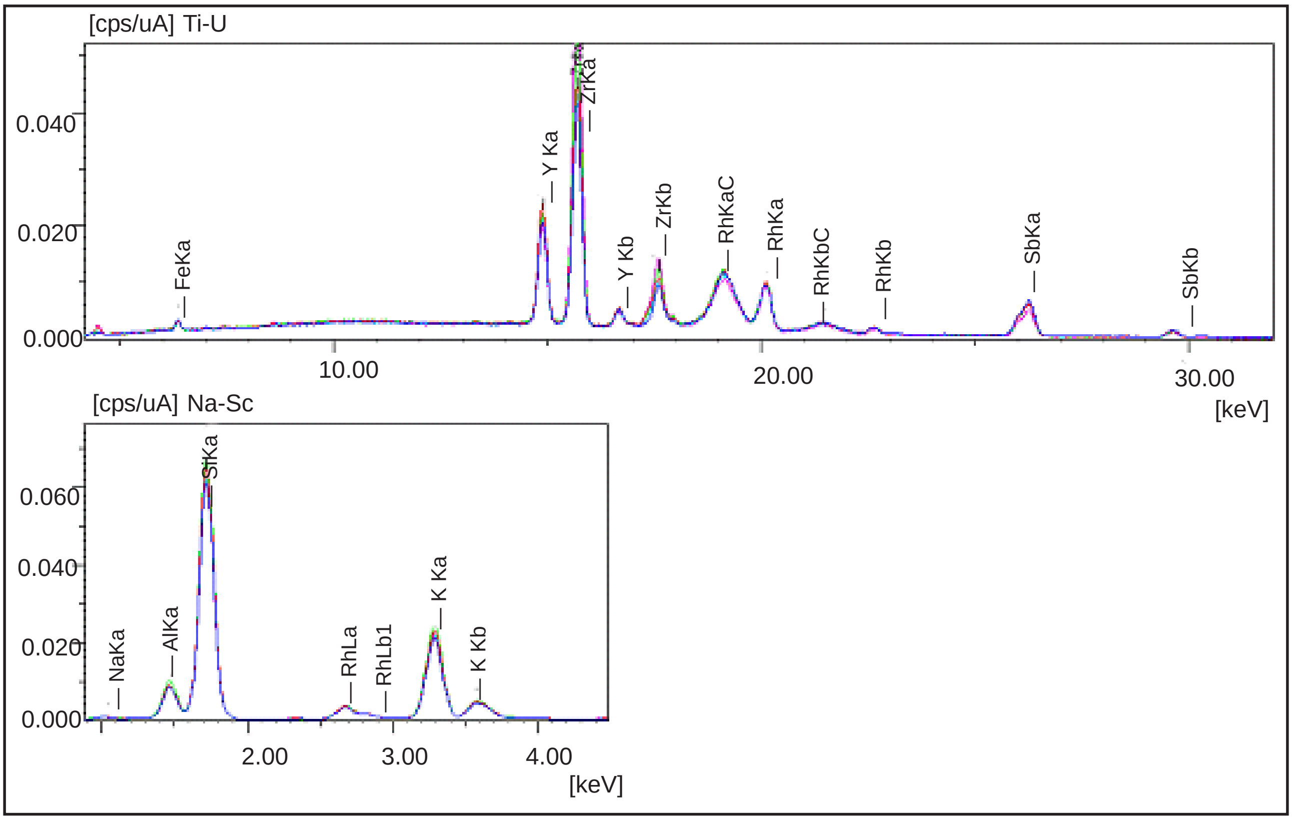

Fig.3: *Overlapped profiles of five porcelain crowns*

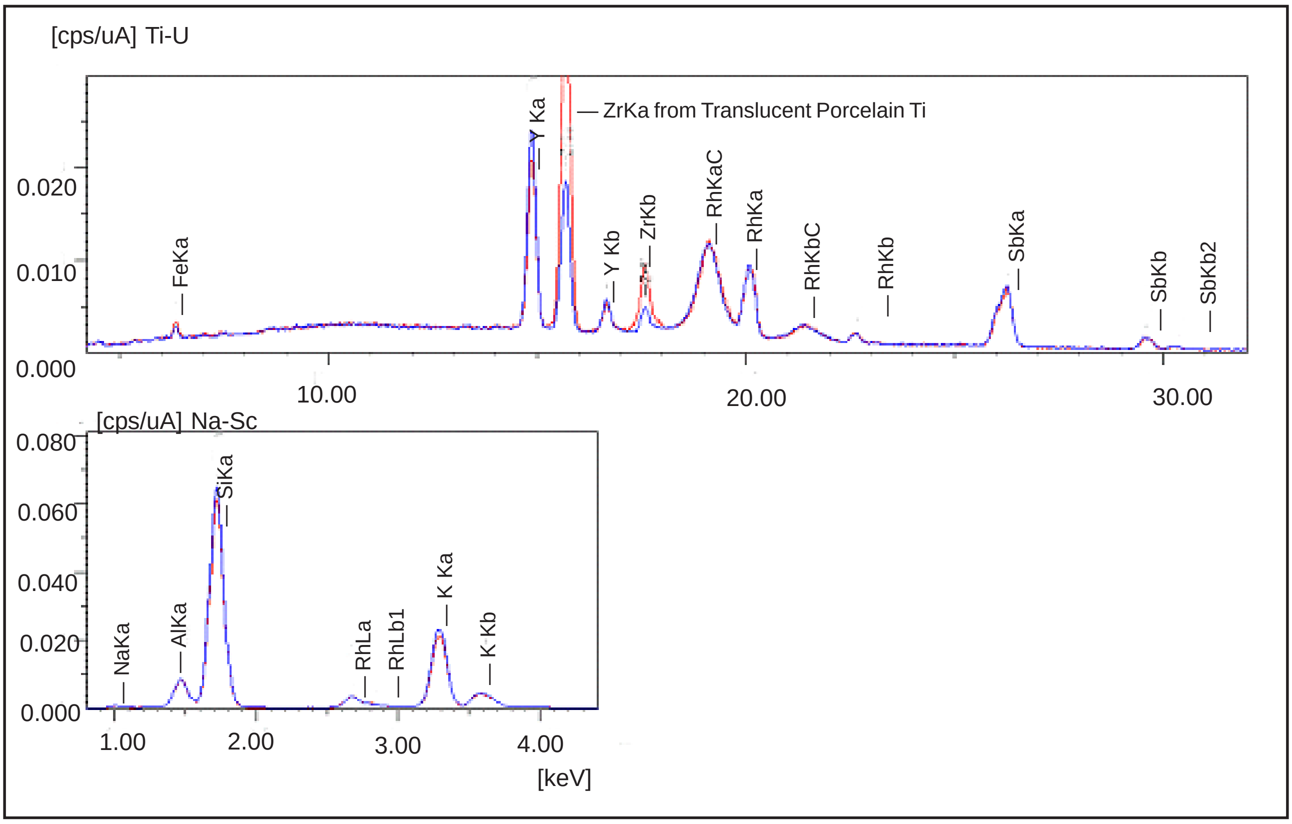

Fig.4: *Overlapped profile of porcelain crown and Porcelain* T_1

qualitative analysis for positive identification of manufacturers/porcelain samples was carried out for each crown by comparing the energy peaks of elements. The target portion of the porcelain crown for this analysis was the center of the labial surface.

RESULTS

A total of 14 elements (Si, Al, Ca, Mg, K, Na, Sb, Zr, Ce, Fe, Ti, S, P and Y) were detected in the porcelains selected, and the compositions were diverse and specific to the different kinds of porcelain (Table 2). Each porcelain contains, by weight, approximately 65-67% Si, 14-15% Al, 10-12% Na and 9-10% K. Differences in the presence of particular trace elements, namely Cerium and Zirconium, were also detected. Cerium was detected only in Tx porcelain powder (0.07%) and no Zirconium was detected in T_0, Tx and T Blue porcelain powders. Fig.3 shows the overlapped profiles from the five crown samples. The profiles from all porcelain crowns analyzed show the same peak patterns at each element energy level. The same element composition could be confirmed on the display. The profile from one of the five was then compared and overlapped with each of the other porcelain powders. The peaks in the spectrum from the crown samples were most similar to that from Translucent Porcelain T_1 (Fig.4), apart from a gap of the peak at the energy level of Zr.

DISCUSSION

Dental porcelains are appreciated as highly esthetic restorative materials with optimal properties that better simulate the appearance of the natural dentition. For the creation of the crown form a dental technician uses different kinds of porcelain powders (for opaqacity, dentin, enamel, incisal detail and translucency) in varying ratios and compositions to make artificial teeth reproduce as closely as possible the natural dentition. Manufacturers provide their own types of porcelain powders to represent the individual natural shade and variations exist between products.

Generally, to specify the shade of porcelain crown to be constructed, a shade guide is used to compare with the patient's remaining natural teeth. Apart from any special requirements in molding or staining, once a dentist has specified a shade, a dental technician will use a variety of porcelain powders to reproduce the same tooth shade according to the manufacturer's instructions. It is reasonable to

Table 2: *Composition of the dental porcelain powder by EDXRE analysis*

	Oxide	Composition (wt. %)													
		E_1	E_2	E_3	T_0	T_1	T_2	T_x	LT_0	LT_1	T•B	S•B	C•E	C•W	I•A
1	SiO_2	67.49	67.32	67.0	67.27	65.88	65.44	64.54	64.95	65.37	66.25	66.27	65.22	62.66	65.03
2	Al_2O_2	15.17	15.25	15.74	15.60	15.70	16.08	14.98	15.84	15.79	15.84	15.63	15.54	15.66	15.52
3	CaO	1.26	1.28	1.32	1.34	1.27	1.30	1.49	1.56	1.63	1.71	1.42	1.71	1.58	1.52
4	MgO	0.11	0.15	0.15	0.13	0.15	0.15	0.26	0.11	0.11	0.12	0.18	0.17	0.12	0.17
5	K_2O	9.15	9.22	9.61	9.29	9.39	9.14	10.38	9.56	9.46	9.64	9.16	9.25	9.00	9.30
6	Na_2O	11.16	11.64	11.40	10.99	11.06	10.35	12.58	11.40	11.15	11.30	12.08	11.65	11.18	10.78
7	Sb_2O_3	1.60	1.64	1.63	1.65	1.56	1.60	1.90	1.70	1.68	1.70	1.64	1.60	1.36	1.58
8	CeO_2	-	-	-	-	-	-	0.07	-	-	-	-	-	-	-
9	ZrO_2	0.33	0.28	0.31	-	0.17	0.31	-	0.04	0.06	-	0.24	0.68	2.89	0.41
10	Fe_2O_3	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04	0.04
11	TiO	0.03	0.03	0.03	0.03	0.03	0.03	0.02	0.03	0.03	0.03	0.03	0.03	0.03	0.03
12	SO_3	-	-	-	0.03	0.02	-	0.03	0.02	-	0.02	-	-	-	-
13	P_2O_5	0.08	0.05	0.04	0.04	0.16	0.23	0.10	0.33	0.33	0.26	0.16	0.58	1.94	0.33
14	Y_2O	0.11	0.11	0.11	0.18	0.17	0.17	0.08	0.16	0.14	0.13	0.15	0.12	0.10	0.12

hypothesize that every powder may contain different substances. It means the identification of porcelain by the elemental composition differences among porcelains may bring out helpful information for identifying, firstly the porcelain manufacturer, and ultimately the unknown person.

Manufacturers are reluctant to provide complete information about the composition of their materials, including porcelains, as they consider this to be privileged information and do not wish their competitors to be aware of this information. We are aware of no published reports that describe, officially, a list of identity and composition of materials for forensic use. This study was undertaken to commence compilation of a database on the elemental composition of dental porcelain materials in Japan. Accordingly, for this preliminary study, we selected one manufacturer only and the shade of Vita-A3, which is a relatively popular shade for Japanese anterior teeth. According to the instruction in the manufacturers' guide for reproducing shade A3, a total of 14 designated porcelain powders were collected and measured for this study.

Silicon, Aluminum and Potassium were found to be the most dominant elements in the porcelain powders at similar concentrations, being virtually indistinguishable from one another in each porcelain powder. These elements might be derived from the main ingredients of potassium feldspar that is considered to be a raw material of dental porcelain. It is, therefore, the presence or compositional variation of other elements that provide the differences among dental porcelain manufactures. In the porcelain product tested, the presence or compositional weight variations for cerium and zirconium may be the characteristic elements.

Thus, there are local compositional variations of the element's present in the different porcelain powders even from one manufacturer. It will, therefore, be necessary to analyse, more dental porcelain powders normally used in crown construction in Japan to ensure reliability of the database. Further extremely accurate quantitative analysis by calibration curve method for each element will be needed to obtain reliable results for the comparative study of the composition of other trace elements present at concentrations of less than 1%.

To confirm the reliability of this method, the spectrum profiles by qualitative analysis for five porcelain-fused-to-metal crowns (Vita shade A3) produced by different dental technicians belonging to different laboratories were compared with each other. Their spectrum patterns were also evaluated against those from 14 different porcelain powders. It was correctly confirmed that the Translucent Porcelain T_1 was contained in the surface porcelain of the sample crowns.

This preliminary study has shown that surface analysis by EDX may have great potential for identifying the composition, and thereby the manufacturers of dental porcelain, which in turn may assist dental identification.

CONCLUSIONS

The dental porcelains examined were composed of 14 kinds of elements, and showed variation in the presence of some trace elements. By confirming the overlapped profile images obtained from the porcelain crowns and porcelain disc samples, it was possible to identify the porcelain manufacturer. Further study to analyze other manufacturers or other types of porcelains and searching for additional effective parameters for identifying individual porcelain powders will be needed for forensic case application.

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REFERENCES

1. Brown KA. The identification of Linda Agostini. Am.J.Forensic Med Pathol 1982;3:131-41.

2. Midda MA. disclosing solution for synthetic filling. Br Dent J 1969;128:519-20.

3. Benthaus S, Duchesne A, Brinkmann B. A new technique for the postmortem detection of tooth-coloured dental restorations. Int J Legal Med. 1998;111:157-9.

4. Wozniak WT, Moore BK. Luminescence spectra of dental porcelains. J Dent Res 1978;57: 971-4.

5. Carson DO, Orihara Y, Sorbie JL, Pounder DJ. Detection of white restorative dental materials using an alternative light source. Forensic Sci Int 1997; 88:163-8.

6. Clark DH, Ruddick RF. Postmortem detection of tooth colored dental restorations by ultra violet radiation. Acta Med Leg Soc. 1985;35:278-84.

7. Pretty IA, Smith PW, Edgar WM and Higham SM. The use of quantitative light-induced fluorescence (QLF) to identify composite restorations in forensic examinations, J Forensic Sci.2002;47:831-6.

8. Suzuki K, Hanaoka Y, Minaguchi K, Inoue M, Suzuki H. Positive identification of dental porcelain in case of murder. Jpn J Legal Med 1991;45:330-40.

9. Yoshida H, Takei T. Identification of artificial teeth by fluorescent examination with an emphasis on metal bond porcelains. Jpn J Legal Med 1993;47:310-4.

10. Aboshi H, Takahashi T, Komuro T, Nogami T. Identification of the unknown body by component analysis of dental porcelain. Acta Crim Japon 2004;70:120-7.

11. Herglotz HK, Birks LS. eds. X-ray spectrometry, New York: Marcel Dekker, 1978.

12. Blakeslee RW, Renner RP, Shiu A. eds. Dental technology - Theory and practice -, St. Louis: CV Mosby, , 1980;224-53.

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