



Analysis of Sakhalin-Ainu lacquerwares by pyrolysis gas chromatography/mass spectrometry

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ABSTRACT

Six pieces of Sakhalin-Ainu lacquerware were analyzed by pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS) to determine the lacquer source. In the direct Py-GC/MS, 3-heptylphenol (P7) and 3-pentadecylphenol (P15) were detected in the mass chromatograms at $m/z = 108$, and palmitic acid and stearic acid were detected at $m/z = 60$ in the mass chromatograms of all pieces of Sakhalin-Ainu lacquerware, implying that drying oil was added to sap collected from a *Toxicodendron vernicifluum* lacquer tree to prepare the lacquer. The energy dispersive X-ray fluorescence results showed that the component ratio of base ground powder was similar to that of Aizu and Joboji lacquerwares, and the Fourier transform infrared spectroscopy results showed that Sakhalin-Ainu lacquerwares used persimmon tannin in the base ground powder like Joboji lacquerwares, suggesting that Sakhalin-Ainu lacquerwares were produced in Joboji, Japan. Based on these results, the production district, history, and culture of the Sakhalin-Ainu lacquerwares are discussed.

1. Introduction

Lacquer is a natural paint that is excellent in durability, water resistance, and chemical resistance; it is also a natural paint that human beings have been using for centuries (Qin et al., 1995). Due to its extremely strong resistance and excellent aesthetic appearance, it is mainly used for the protection of crafts, as an adhesive, and decoration. Therefore, lacquer is used for many important historical crafts and cultural assets. In addition, even in areas where lacquer was not used, there are cases where imported lacquerware was used (Delphine, 2016; Hagelskamp et al., 2016; Körber et al., 2016).

Sakhalin-Ainu lacquerware refers to the lacquerware that the Ainu people imported from Japan (Prokofiev, 2005; Kitano, 2014; Asakura, 2016; Tanimoto and Asakura, 2016). What kind of history Ainu peoples had and how they passed through to early modern times are still unclear. The Ainu culture grew and developed from northern Iwate prefecture to southern Sakhalin around the 17th and 18th centuries A.D. (Vanstone, 1993; Koji, 2014). In this period, trade was actively carried out, and lacquerware was imported from Japan (Koji, 1997). However, it is unclear where in Japan the imported Sakhalin-Ainu lacquerware was produced. Therefore, we attempted to investigate the site of the Sakhalin-Ainu lacquerware scientifically to identify the area where trading was done at that time and contribute to the Ainu historical investigation.

In this study, we clarified the materials used for production of the

lacquer variety used in the six pieces of Sakhalin-Ainu lacquerware. For analysis of the lacquer, pyrolysis-gas chromatography/mass spectrometry (Py-GC/MS) was carried out because it requires only a small amount of sample and does not require sample preparation (Lu et al., 2007; Wei et al., 2011a, 2011b; Karpova et al., 2017; Sung et al., 2016). In addition, cross-section observation, attenuated total micro Fourier transform infrared spectroscopy (micro FT/IR), and energy dispersive X-ray fluorescence (ED-XRF) were performed. In addition, six Sakhalin-Ainu lacquerwares were analyzed in comparison with Japanese lacquerwares.

2. Experimental

2.1. Materials

Six pieces of ancient lacquerware belonging to the Sakhalin Regional Museum of Local Lore, Institute of Heritage Bronislaw Pilsudski, Russia, were analyzed in this study. The samples are shown in Fig. 1, and the detailed information is described below.

Sample 1: Museum collection number No.2270-15-438. It is a big cup with a mouth that is 22.3 cm inside diameter and 23.2 cm outside diameter. The height is 16.3 cm, annular support leg is 12.6 cm, and height is 4.1 cm, respectively.

Sample 2: Museum collection number No. 2270-31. It is a large heating

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Fig. 1. Photos of six Sakhalin-Ainu lacquerware samples.

- vessel that was produced after the Meiji period.
- Sample 3: Museum collection No. 2270-59. It is a vessel for pouring liquor.
- Sample 4: Museum collection No. 40-15. It is a cup made at the end of the Tokugawa period using conifer wood as the body.
- Sample 5: Museum collection number No.40-5. It is a round pot with an inside diameter of 30 cm. The production period is considered to be the late Edo period.
- Sample 6: Museum collection number 231. It is a large-sized bowl with a drawn tray pattern drawn.

The Edo period in Japan is 1603–1867 years, Meiji era 1868–1967 years. The Sakhalin-Ainu lacquerware analyzed this time was found to be from the carbon 14 radioactive dating to the end of the Edo period.

2.2. Cross section observation

A cross section of the sample was analyzed by the thin section method. Samples were first sandwiched between sample clips (Buehler ITW Co., Ltd.) and embedded in epoxy resin (Pelnox Co., Ltd., Japan). Then, the surface was polished using diamond powder (Buehler #400, #600, #800). The prepared sample had a thickness of 10 to 15 μm . Finally, the sample layer was observed using an optical microscope (ECLIPSE LV100N POL, Nikon).

2.3. Pyrolysis-gas chromatography/mass spectrometry

Py-GC/MS measurements were carried out using a vertical micro-furnace pyrolyzer PY-3030D (Frontier Lab, Japan), a Hewlett-Packard 6890 gas chromatograph, and a HPG 5975MSD (Hewlett-Packard, Ltd.) mass spectrometer, as previously reported. The ionization energy (EI mode) was 70 eV, and the sample was scanned from $m/z = 29$ to $m/z = 800$ at 1 Hz. Gas chromatography/mass spectrometry was

controlled by chromatogram analysis software and data were analyzed with Agilent MSD Chemstation software. All the product compounds were identified with reference to the NIST mass spectrum library.

2.4. Energy dispersive X-ray fluorescence

ED-XRF measurement performs elemental analysis by X-ray irradiation of a sample. This method reveals the elements of sample decoration. In this study, the elements of the cross section were analyzed by ED-XRF with an XGT-5200 series instrument (Horiba, Co., Ltd., Tokyo, Japan). The tube voltage and current were set to 50 kV and 1.0 mA, respectively.

2.5. Attenuated total reflection Fourier transform infrared spectroscopy

Micro FT/IR was performed using a Nicolet iN 10 instrument (Thermo Fisher Scientific Co., Ltd., Japan). The measurement range was 675–4000 cm^{-1} , the resolution was set to 8 cm^{-1} , and the attenuated total reflection estimation frequency was 128 Hz using a Ge tip.

3. Results and discussion

3.1. Py-GC/MS

Fig. 2 shows the Py-GC/MS results at $m/z = 108$ of samples 1 (Fig. 2A) and 2 (Fig. 2B). Both samples 1 and 2 showed a mountain-like peak. Compared with the characteristic products of standard lacquers, both these two samples have the characteristic peaks of urushiol appearing at 3-heptylphenol (P7) and 3-pentadecylphenol (P15). Samples 3, 4, 5, and 6 also showed Py-GC/MS peaks at $m/z = 108$, similar to samples 1 and 2, suggesting that all six Sakhalin-Ainu lacquerware samples were coated with sap collected from *Toxicodendron vernicifluum* lacquer trees, which grow mainly in Japan, China, and Korea.

When the red color on samples 2, 5, and 6 (Fig. 1) was analyzed, the

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