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Analysis on Quality and Composition of Ink Pigments Used for Semi-permanent Makeup

Hyun-Sook Jin[1], Byung-Soo Chang*[2]

[1]*Researcher, Department of Cosmetology, Hanseo University, Seosan, Chungnam 31962, Republic of Korea*

[*2] *Professor, Department of Cosmetology, Hanseo University, Seosan, Chungnam 31962, Republic of Korea*

kangsj0522@naver.com[1], *bschang@hanseo.ac.kr*[*2]

Corresponding author[*]: mobile Phone: +82-10-6279-0810

Abstract

Background/Objectives: **Background/Objectives**: This study selects some brown pigments manufactured by both local and foreign companies and analyzes their ultrastructural features and element compositions, in comparison of inorganic substance included in each pigment to define a possible problem and the cause after a semi-permanent makeup.

Methods/Statistical analysis: A scanning electron microscope (SEM) is employed to see the form and microstructure of each pigment added to the local and foreign ink products selected, and an energy dispersive x-ray spectrometry (EDX) analyzes the element composition.

Findings: The pigment from abroad is composed of iron (Fe) and titanium (Ti). The SEM tells it is made of iron oxide and titanium dioxide. The local ink consists of iron (Fe), manganese (Mn), silicon (Si), aluminum (Al), and sodium (Na). Observation on the SEM finds that characteristically there are a multiple micrometer-sized of polygonal flat substance and lots of impurities.

Improvements/Applications: It is found that differences in homogeneity, roughness, and size of pigment particles are largely varied depending on whether the ink is expensive or not. It is considered that the pigment's heterogeneity or an addition of contaminant, intentional or accidental, will have a big effect on customer satisfaction after a semi-permanent makeup procedure.

Keywords: EDX, Iron oxide, Semi-permanent makeup, Tattoo, Titanium dioxide, SEM.

1. Introduction

Semi-permanent makeup such as having an eyebrow or lip line tattoo for beauty is increasingly popular among adults from all ages around the world[1]. There are art tattoos and medical tattoos. The former is to decorate your body expressing an artistic sense, and the latter is to hide scars or shortcomings in the skin[2,3]. A medical tattoo is used to make nipples look like real after damage or surgery, or to treat a birthmark, alopecia areata and vitiligo. It also can be used to complement makeup for patients having an allergy to cosmetics[4]. For semi-permanent makeup for beauty, a tattoo artist would puncture the skin with a sharp needle and inject the ink into the superficial layer of dermis by hand. It will last for six months to three years.

There are three tattooing techniques: embroidery technique, hand acupuncture technique, and machine technique. The number, length, and shape of the needles are differentiated according to the technique you choose. In the embroidery technique, multiple needles which look like knife blades in naked eye scratch the skin creating diagonal lines to inject ink into the deep layer of the skin. In the hand acupuncture technique, more than three needles puncture or take off the skin to inject ink producing a three-dimensional effect.

The machine technique punctures the skin moving needles up and down powered by a motor, which is launched in makeup machine, to inject ink into the skin. You may see blood since the procedure lasting for minutes or longer, not exceeding an hour and you will experience crusting and swelling in the affected area for about two to five days. Then, the skin will be healed. As an after effect, however, you may suffer allergy symptoms and granuloma or have keloid caused by an excessive growth of collagen fibers leading to a scar. It is also known that microblading or lash enhancement tattoo may cause necrosis on the eyelid[5].

The Korea Consumer Agency announced that the Consumer Injury Surveillance System (CISS) have received 77 reports from consumers on damage by a semi-permanent makeup treatment for three years and six months from Jan 1,

2013 to June 2016. The biggest complaint is side effects after the procedure (55 cases or 71.4%), followed by 16 cases or 20.8% complaining about negligence during the procedure like ink or anesthetic in the eye and 6 cases or 7.8% expressing dissatisfaction of the procedure. Tattoo inks released in the market include barium (Ba) and carcinogens such as aromatic amine. They are easily absorbed inside the body damaging the muscles, kidney, and heart. They also cause arrhythmia, paralysis, and gastrointestinal changes intoxicating the body. Therefore, substances including barium have been withdrawn from the market (https://www.ciss.go.kr/www/index.do).

Amid an increase in side effects after a semi-permanent makeup treatment, studies on quality and composition of inks are increasingly being reported[6]. Still, however, studies about whether inks, which are widely used for semi-permanent makeup, have heavy metals by analyzing the composition are not enough.

Thus, this study is geared towards identifying a possible problem and the cause after a semi-permanent makeup treatment and analyzes composition of brown pigments, one of the most widely used in the industry.

After selecting the most popular local and foreign ink products, the study identifies the form and feature of particles added to the products using scanning electron microscope and analyzes the composition of inorganic substances with energy dispersive X-ray spectrometry.

2. Materials and Methods

2.1. *Materials*

To analyze the ultrastructure and composition of particles of brown pigments used for semi-permanent makeup a single type of lower-priced local product (E Co., brown color) and higher-priced foreign product (V Co., light brown color) each were selected for experiment.

2.2. *Methods*

To separate the pigment in ink for both local lower-priced and foreign higher-priced products, researchers placed 1 ㎖ of each ink sample in falcon tubes and added absolute alcohol for each. The samples were diluted in centrifuge and left to make a precipitation reaction. Researchers removed the organic supernatant liquid and ran through the same process three more times.

2.2.1. Observation Using Scanning Electron Microscope (SEM)

Each sample was mounted on a carbon-taped stub and naturally dried then coated with platinum (20 nm) using an ion coater (IB-5 ion coater, Eiko, Japan), to see the type of inorganic material inside the inks and their ultrastructure. An SEM (S-4700, Hitachi, Japan) was used at 15 kV for observation.

2.2.2. Analysis by Energy Dispersive X-ray Spectrometry (EDX)

To compare and analyze composition of inorganic material added to the inks, each group (lower-priced and higher-priced products) of specimen was collected and mounted on a copper-taped stub and dried in a vacuum drying oven at 60℃ (Yamato, DPF-31, Japan) for 24 hours, then coated with platinum (20 nm) using an ion coater (IB-5 ion coater, Eiko). The coated specimen was analyzed using an EDX spectrometry (INCA, Oxford Ins, Great Britain) at acceleration voltage of 15 kV.

3. Results

A lower-priced local semi-permanent makeup pigment was observed by SEM. As figure 1 shows, micro-pigment particles in the specimen observed by an SEM in low magnification are evenly spread but there exist some bigger flat material with higher electron density among microparticles as well [Figure 1].

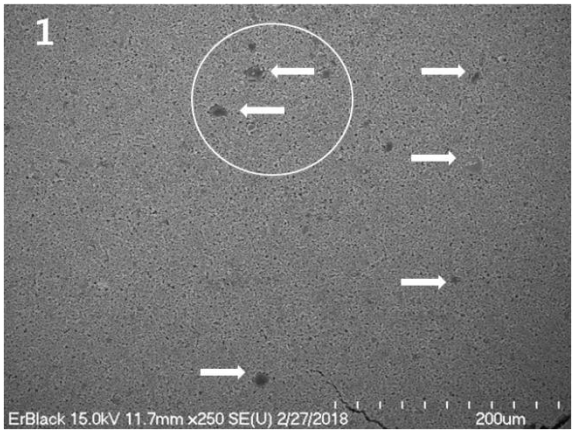

Figure 1. Image of pigment added to lower-priced ink for semi-permanent makeup observed by SEM in low magnification. Arrows point out flat material and the circle shows the enlarged image in the figure 2.

The flat material is polygonal and its end is pointed or angular. It is multi-layered and the surface looks smooth. Cubic-shaped or rod shape microparticles in addition to the flat matter are also easily observed. The polygonal flat

matter measures about 10 ㎛ in diameter and nano-unit iron oxide is dispersed in the surroundings [Figure 2].
In order to identify the pigment composition of the ink researchers determined the area to be analyzed based on the microscopic data. The spectrum as the following picture shows was analyzed by EDX [Figure 2].

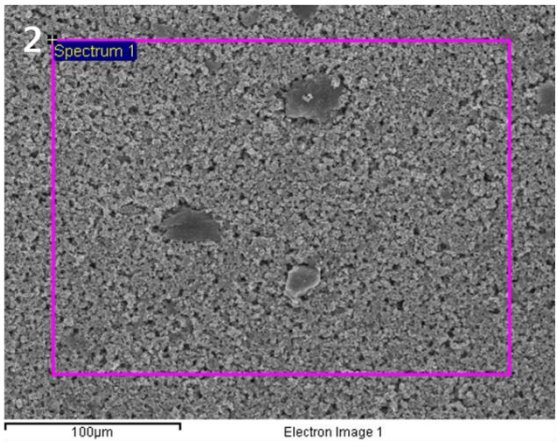

Figure 2. Picture of the spectrum for EDX analysis of the pigment (lower-priced ink for semi-permanent makeup) specimen observed by SEM

As a result of the EDX analysis on the pigment composition of lower-priced semi-permanent makeup ink, iron (Fe), manganese (Mn), silicon (Si), aluminum (Al), and sodium (Na) were detected. Excluding carbon (C) and oxygen (O) the composition is a mixture of iron (31.61%), manganese (0.46%), silicon (1.18%), aluminum (0.50%), and sodium (0.57%) [Figure 3].

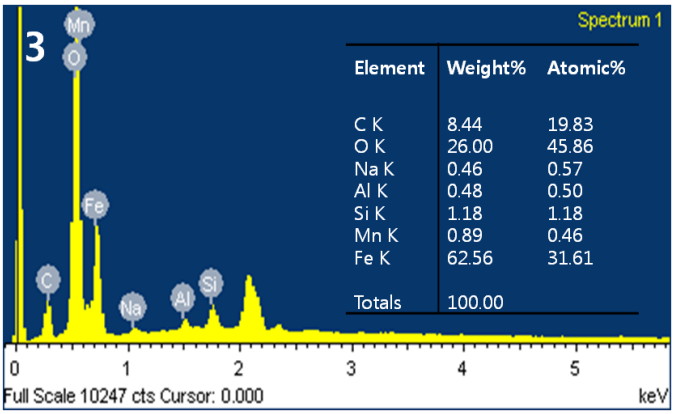

Figure 3. Analysis on lower priced semi-permanent makeup ink by the energy dispersive X-ray spectrometry showing detection of elements, such as iron (Fe), silica (Si), manganese (Mn), aluminum (Al) and sodium (Na)

Figure 4 demonstrates a mix of cubic- and rod shape iron oxides and flat matters in the ink. It is notable to see some flat material measuring about 0.5 ㎛ to 1 ㎛ in diameter among metal elements.
Figure 5 shows about 250um of cubic-shaped black iron oxides observed by a SEM in high magnification.

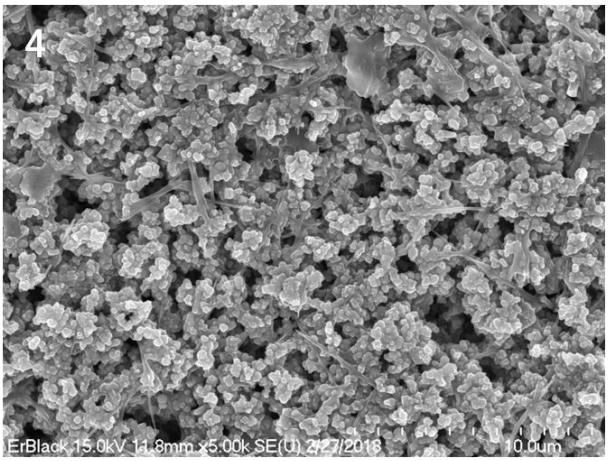

Figure 4. Round black iron oxides in the lower-priced semi-permanent makeup ink observed by scanning electron microscope.

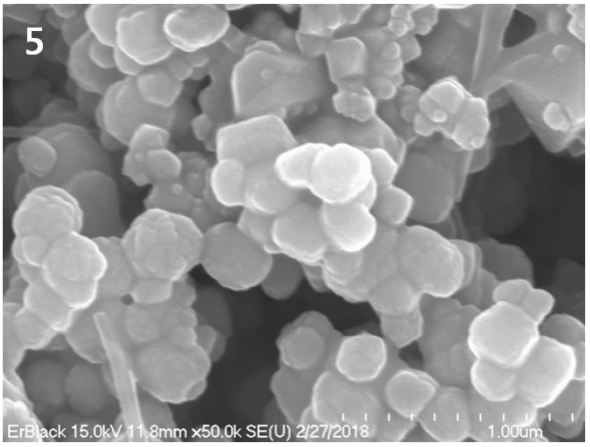

Figure 5. Black iron oxides in the lower-priced semi-permanent makeup ink observed by a scanning electron microscope in high magnification.

Figure 6 is the electron microscopic image in low magnification of pigment in higher-priced semi-permanent makeup ink. It shows that metal elements in the ink are evenly spread and some microparticles with the same shape are held together. Unlike the previous lower-priced product, the present ink does not have any flat matter or impurities [Figure 6].

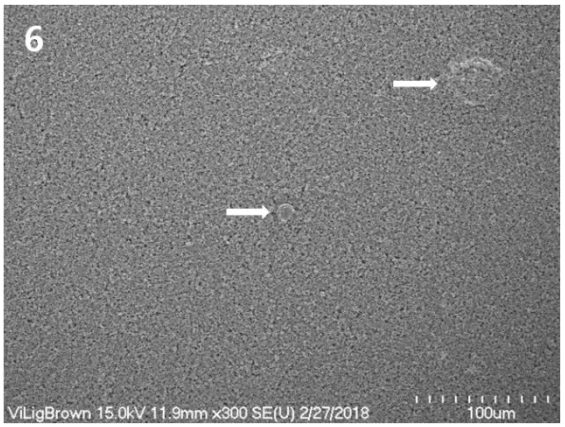

Figure 6. Scanning electron micrograph in low magnification of pigment in higher-priced semi-permanent makeup ink which is highly homogeneous. Arrows point out chunks of iron oxide.

Looking at some chunks of metal elements from figure 6 in a higher magnification, rod shape iron oxides are held together due to electrostatic attraction [Figure 7]. There exist titanium dioxides surrounding the chunks. Most chunks

of iron oxide look round or globular measuring about 5 ㎛ in diameter [Figure 7].

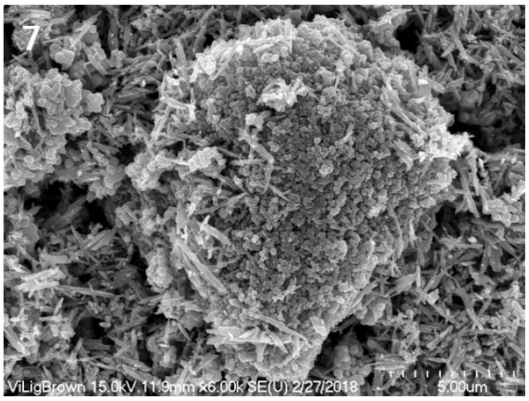

Figure 7. Scanning electron micrograph of a chunk of iron oxide observed in higher-priced semi-permanent makeup ink pigment.

In order to identify pigment composition of the higher-priced ink, the spectrum to review was scanned and determined to analyze by EDX, and produced the microscopic image as is seen from figure 8. It has been assumed that there are rod shape iron sulfate and red iron oxide, cubic-shaped black iron oxide, and titanium dioxide. Other small cubic-shaped particles are also found [Figure 8].

As a result of the EDX analysis of the spectrum seen in figure 8, iron (Fe) and titanium (Ti) were detected but no other metal elements [Figure 9]. The composition is a mixture of iron (31.66%) and titanium (3.35%) except carbon and oxygen [Figure 9].

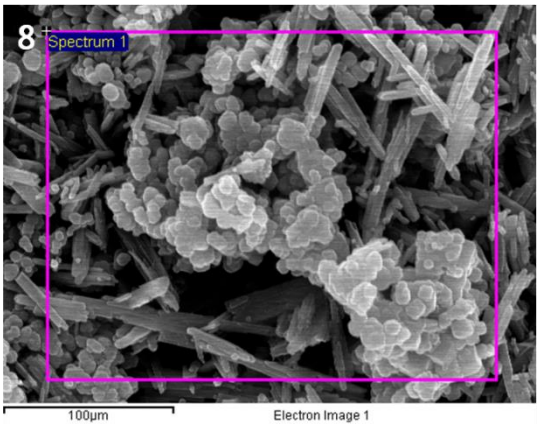

Figure 8. EDX analysis image for the spectrum, which is given by the scanning electron microscopic data for higher-priced semi-permanent makeup ink specimen.

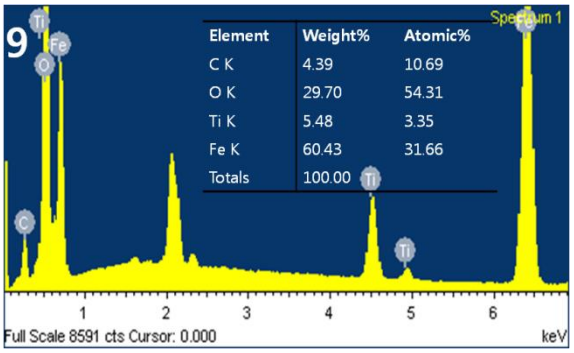

Figure 9. Analysis on higher priced semi-permanent makeup ink by EDX detecting iron (Fe) and titanium (Ti).

Figure 10 is an image of the foreign brand semi-permanent makeup ink pigment in high magnification observed by SEM. Each different metal element is combined among themselves with the same shape. Rod shape iron oxides come

in different sizes and easily distinguished from the cubic-shaped black iron oxides and titanium dioxides. However, you could not tell iron sulfates and red iron oxides from a mixture of iron oxides only based on the length of the rods [Figure 10].

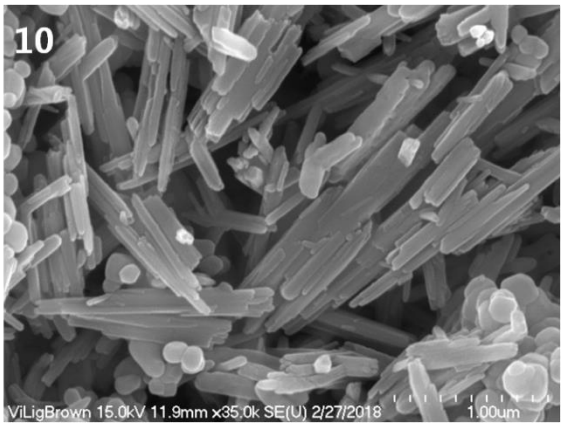

Figure 10. Image in high magnification of rod shape iron oxides and cubic-shaped titanium dioxides added to the higher-priced semi-permanent makeup ink pigment observed by scanning electron microscope.

4. Discussion

Tattooing is inserting exogenous ink pigment into the dermis of the skin[7] to change its color permanently. Tattoos are forms of body art that belong to a multitude of different cultures and civilizations all over the world. Tattoos date back to even before ancient Egypt[2]. Cosmetic tattooing, or semi-permanent makeup, began a trend since the late 1970s helping make the face symmetrical or make a better look [8, 9]. Some of the most popular tattoo placements are eyebrows, eye lines, lip lines, whole lips, cheeks, and facial spots for cosmetic value[2,8].

The pigment used for ink is a water insoluble inert substance classified as a cosmetic ingredient or color additive complement[2]. In the manufacturing process of the tattoo ink the color appears differently depending on how much metal elements were added and how much the metals were refined[10].

A semi-permanent makeup ink is a mixture of water in which a water soluble pigment is dispersed and it includes dispersants, preservatives, and fragrances. Among them, the water soluble colorant is a determinant factor consisting of coloring material like carbon black, titanium dioxide, and iron oxide as well as auxiliary material[11].

The present study found that there was a big difference in composition and quality between lower-priced and higher-priced ink pigments. While there are large-sized (ranging from some micrometers to tens of micrometers) impure particles were found in the lower-priced ink pigment, no other metal elements than iron oxide and titanium dioxide were found in the higher-priced pigment. Jin & Chang[6] explained that the particle size and composition differ depending on impurities added by accident of on purpose.

The ink pigment is mainly composed of a sub-micrometer of iron oxide and titanium dioxide. The color varies according to the mix proportion among iron sulfate, black iron oxide, and red iron oxide[6,12]. Some metal oxides are intentionally used to obtain a certain color and they may give rise to negative effects and complications in the human body [13].

From the observation by a scanning electron microscope of the present study, dozens-of-micrometers of particle aggregation was found in iron oxides in the higher-priced semi-permanent makeup ink forming a tiny chunk. But there was no aggregation in the lower-priced one. It is assumed that the difference occurs due to a different proportion of iron oxides or an existence of impure particles.

Iron oxide features an easy aggregation because of high surface energy level [14]. Titanium dioxide is widely used in paint, textile, paper, plastics, sun screen, cosmetics, toothpastes, surface coating, and food additives as a commonly used nano particle [15].

This study which analyzed lower- and higher-priced ink pigments by EDX detected iron (Fe), manganese (Mn), Silicon (Si), aluminum (Al), and sodium (Na) from the lower-priced one and iron (Fe) and titanium (Ti) from the higher-priced one.

The major component of pigments used for tattoo is metal. Tattoo inks may contain up to 10% of impurities as they are usually manufactured in the industrial process like varnish and printing [16]. Nonetheless, dichromate Co, dichromate CD, and Hg-salt which may be carcinogenic and cause skin sensitization are restricted from adding although they are basic pigments of green, blue, yellow, and red colors. On the other hand, iron oxide, carbon, and manganese are commonly used for brown, white, black, and purple colors. A specific color can be created by mixing organic colors, aluminum (Al), calcium (Ca), and cadmium (Cd) with different brightness [13].

Forte et al.[13] analyzed 56 tattoo inks from 4 different manufacturers using ICP-MS to see the presence of heavy metals inside. Generally, metal elements added to tattoo inks are Al, Ba, Cu, Fe, and Sr. As a result of the analysis, Al,

Ba, Cd, Co, Cr, Cu, Fe, Hg, Mn, Ni, Pb, Sb, Sr, and V were detected, they said. Among them, Cr, Ni, and Co which may cause allergic reactions were added too much and were exceeding the standard level of 1μg / g and toxic metals like Cd, Mn, Pb, and V also reached more than 1μg/g.

In a similar context, the present study also detected manganese from the lower-priced ink pigment which is an allergy ingredient possibly affecting the human body. If the skin is in contact with a tattoo ink for a long time more than one metal elements in the pigment cause allergic reactions, eczema, lichenoid, gastric lymphoma, or granuloma[17-20].

As earlier said, Si, Al, and Na were detected from the lower-priced pigment specimen of this study. These elements, not detected from the higher-priced one, are assumed to have intentionally been added to give a feature of viscosity, brightness, and anti-bacterial effect or originated from any impure particle as Arl et al.[21] reported.

Highly refined pigments with high purity should be used to reduce possible harm on the human body. Manufacturing facilities and relevant technologies need to be improved even more to enhance safety levels in tattoo inks[22].

5. Conclusion

The present study analyzed brown semi-permanent makeup ink pigments (lower-priced and higher-priced) using an energy dispersive x-ray spectrometry (EDX) and scanning electron microscope (SEM) to see their compositions and microstructures. For the lower-priced pigment, micro-iron oxide particles in the specimen were observed by an SEM which were evenly spread but there also exist some bigger flat matters and impurities. The polygonal flat matters sized micrometers to tens of micrometers in diameter. As a result of the analysis on the lower-priced pigment specimen by the EDX, Fe, Si, Al, and Na were detected.

The higher-priced ink pigment was composed of rod shape iron oxide, red iron oxide, cubic-shaped black iron oxide, and titanium dioxide. As a result of observation by SEM in high magnification, the iron oxides in a same type formed in a chunk and several groups of chunks were found. In the higher-priced ink pigment, which was analyzed by the EDX, Fe and Ti were detected but no other elements.

6. References

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