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# Preliminary Study of Cosmetic Coloured Contact Lenses Chemical Elements Analysis using Energy Dispersive X-Ray Spectroscopy

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## ABSTRACT

This paper examines the chemical elements used as colour additives in cosmetic coloured contact lenses (Cos-CCL) using Field Emission Scanning Electron Microscope equipped with Energy Dispersive X-ray Spectroscopy (FESEM-EDX) analysis. The samples comprised two different Cos-CCL brands and colours (sample A1-black iris colour & B1-gray iris colour) with their respective clear contact lens counterparts as controls (sample A2 & B2). The parameters of Cos-CCL were observed carefully so that they resembled their respective controls. All the samples were analysed for chemical element characterisation by using EDX spectroscopy surface mapping analysis on both front and back surfaces. EDX spectroscopy point analysis was done on cross-section surface of Cos-CCL when colour additive pattern could not be detected by FESEM on either surface. FESEM-EDX spectroscopy analysis has revealed iron element in the colour additives of the A1 sample and aluminium elements in the B2 sample. These two elements were not present in the respective control samples. It can be concluded that iron and aluminium elements are exclusively attributed to the colour additive in Cos-CCL samples. It is important for manufacturers of Cos-CCL to disclose information of their products and create greater awareness on the risks facing users.

*Keywords:* Colour additives, cosmetic coloured contact lens, elemental analysis, energy dispersive x-ray analysis

## INTRODUCTION

Cosmetic colour contact lenses (Cos-CCLs) is used to change the physical colour appearance of the eyes. They are commonly used as a fashion accessory rather than for refractive correction purpose. Cos-CCL is usually worn by teenagers and adolescents especially in Asian countries (Morgan et al., 2012; Morgan

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et al., 2013). As their popularity increases, many reported cases of complications caused by Cos-CCLs wear have become a public concern. In general, these adverse clinical reports are linked with poor user compliance (Schanzer et al., 1989; Bucci et al., 1997) and acquisition of the Cos-CCL without prescriptions from authorised optical shops (Singh et al., 2012; Steinemann et al., 2003). Thus, it is believed that any adverse contact lens-related ocular occurrence is solely the result of external factors. In fact, none of the studies was found to concisely demonstrate the potential threat from the Cos-CCL materials per se.

A study by Sauer and Bourcier (2011) has reported poor prognosis following microbial keratitis (MK) treatment by Cos-CCL users compared to the control group using clear corrective contact lenses. This discrepancy is probably due to the existence of exclusive Cos-CCL elements used in colour additives despite the presence of a long-standing list of approved colour additives used for Cos-CCL provided by Food & Drug Administration (FDA) of the United States (US).

A recent study on colour additives in Cos-CCL reported the presence of various elements such as iron, chlorine and titanium in Cos-CCL samples (Hotta et al., 2015). However, the study is inconclusive because the researchers were unable to confirm whether the elements belong to the colour additives of Cos-CCL. This study aims to examine the characterisation of chemical elements used as colour additives in different coloured range of Cos-CCL in the presence of their respective clear contact lens counterparts using Field Emission Electron Scanning Microscope equipped with Energy Dispersive X-ray (FESEM-EDX) spectroscopy analysis.

## METHOD

The characterisation of chemical elements in contact lens samples was set up in an experimental laboratory study design. Direct comparison of the chemical elements between Cos-CCL samples and their respective clear contact lens control samples was made. The chemical elements found exclusively on Cos-CCL samples by EDX spectroscopy analysis showed their presence as components of colour additives. The findings were also confirmed by observing the similarities between colour additives patterns of the EDX elemental mappings with the respective FESEM images. To examine the chemical elements in different colour types, the EDX spectroscopy findings were compared between two Cos-CCLs of different colours. The instrument used was Field Emission Electron Microscope (FESEM) JSM-6701F equipped with energy dispersive X-ray (EDX) spectroscopy (JEOL).

### Materials and Instrumentation

**Contact lenses.** Two brands of contact lenses (brand A and B) were studied. These two brands were selected because each brand manufactures both Cos-CCLs and clear corrective contact lenses. The chosen colours were black iris from brand A and grey iris from brand B. The parameters of each Cos-CCL were studied so that they resembled parameters of the respective clear contact lens used as control samples. Brand A contact lens samples (Cos-CCL in black iris colour and its clear contact lens counterpart), are made from etafilcon A. The water content in each sample is 58%. The diameter and base curve on the other hand is 14.2 mm and 8.5 mm, respectively. Brand B contact lens samples (Cos-CCL in grey iris colour and its clear contact lens counterpart), are made from lotrafilcon B with 33% water content in each sample. The diameter of each is 14.2 mm with 8.6 mm base curve. The parameters are listed in Table 1.

Table 1
*The respective parameters of the contact lens samples*

| Brand | Sample | Product name | Colour | Contact lens material | Water content (%) | Diameter (mm) | Base curve (mm) | Colour additive information |
|---|---|---|---|---|---|---|---|---|
| A | A1 | FAE | Mystical black | Etafilcon A | 58 | 14.2 | 8.5 | NIL |
| | A2 (control) | 58F | Clear | Etafilcon A | 58 | 14.2 | 8.5 | N/A |
| B | B1 | AOC | Grey | Lotrafilcon B | 33 | 14.2 | 8.6 | NIL |
| | B2 (control) | AO | Clear | Lotrafilcon B | 33 | 14.2 | 8.6 | N/A |

*Note:* NIL – no information listed; N/A – not applicable

**Field Emission Electron Microscope with energy dispersive X-ray attachment (FESEM-EDX).** The instrument used to characterise the chemical elements in contact lens samples is the Field Emission Electron Microscope (FESEM) JSM-6701F with energy dispersive X-ray (EDX) spectroscopy attachment (JEOL). This instrument works by analysing the X-ray spectrum emitted by the samples bombarded with a focused beam of electrons. Thus, FESEM was used in this study as it has electron probe capabilities. The FESEM was equipped with the EDX spectroscopy to scan the X-ray beam for characterisation of chemical element analysis.

## Protocol

**Preparation of the contact lens samples.** The contact lenses were cut into sizes to fit the scanning electron microscope's stubs. They were cut specifically into approximate quarters by applying single pressure on them using a clean razor blade. Each individual piece was then manually torn and held with a clean tweezer and pat dry, prior to being placed on the FESEM's stub. For each contact lens, two different surface orientations (front surface and back surface) were prepared for analysis.

**Characterisation of chemical element of the contact lens samples**. The stubs containing the samples were placed carefully into the FESEM chamber and examined closely to locate the position of colour additives. Where localisation of colour additives on the sample surfaces were not visible by FESEM, a scanning process was performed on the cross-section surface.

The characterisation of chemical components of the colour additives was performed by EDX surface mapping analysis. The surface of every sample was analysed by EDX surface mapping analysis or EDX point analysis. Summary on the protocol involving FESEM-EDX spectroscopy analysis is shown in Figure 1.

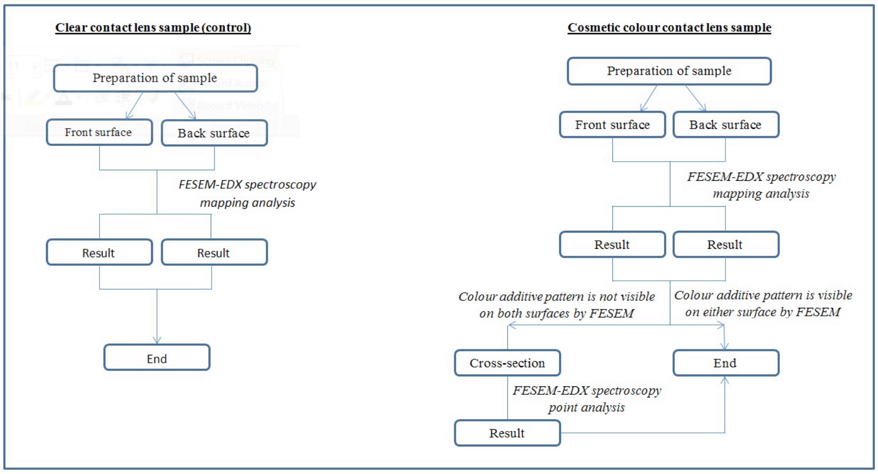



*Figure 1.* Schematic workflow of the FESEM-EDX spectroscopy analysis

## RESULTS

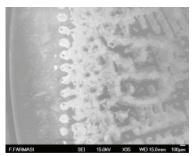
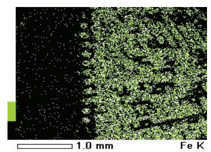
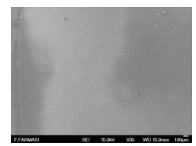
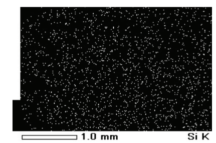
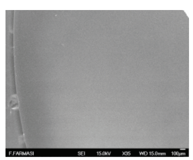
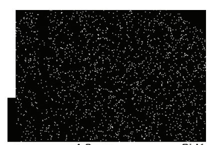
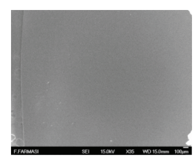
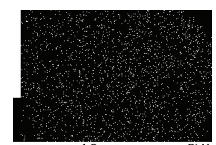
The existence of chemical elements attributable to colour additives of the Cos-CCL samples were established through comparison with the chemical elements found in the respective control samples. Comparison of the chemical elements between two Cos-CCL samples was also done to acquire variation of chemical elements in different colour types. The data in Table 2 and Table 3 feature the manufacture related Cos-CCL samples with their respective control samples.

### Chemical Element Analysis of the Cos-CCL Samples Featuring the Control Samples

As presented in Table 2, chemical elements found by EDX spectroscopy analysis on brand A Cos-CCL sample (A1) were iron, on the front surface and silicon, on its back surface. In contrast, EDX spectroscopy analysis has shown only silicon element on the front and back surface of the control sample (A2). The findings suggested that the element iron is exclusively attributed to the colour additives of the sample. Furthermore, the mapping pattern of iron was concentrated in the area of colour additive portion imaged by FESEM. For the back surface EDX spectroscopy analysis of the same sample found silicon to be diffusely distributed without any specific pattern. These findings were similar to the A2 sample surfaces. Hence, it is suggested that iron is used as a colour additive in A1 sample.

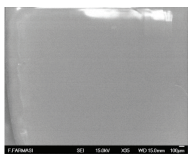
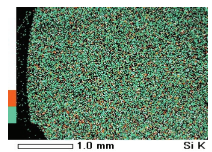
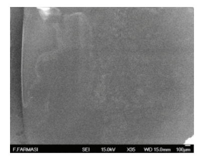
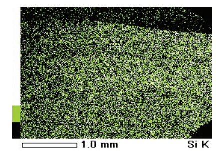
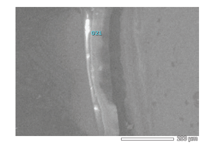
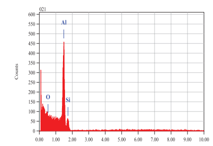
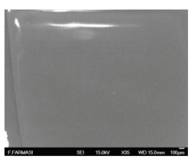
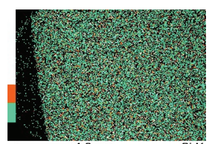
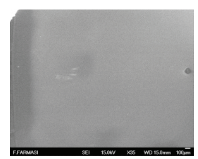
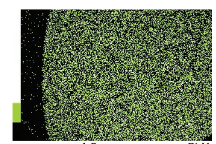
For brand B samples as shown in Table 3, the element of silicon has been analysed on the front and back surface of both Cos-CCL sample (B1) and its control sample (B2). These findings conceded with FESEM image analysis in which the colour additives pattern of the B1 sample was undetected in either surface despite of its visibility by the naked eyes. However, upon performing cross-section FESEM image analysis, the portion of colour additive was found embedded in the matrix of the B1 sample. Hence, the EDX spectroscopy analysis was done by point analysis and revealed the colour additives for B1 sample which is grey in colour contained aluminium.

Table 2
*FESEM-EDX spectroscopy findings of the brand A: Comparison of chemical elements between Cos-CCL samples with control samples*

| Colour / Sample | Surface analysis | | | | | |
|---|---|---|---|---|---|---|
| | Front | | Back | | Cross-section | |
| | FESEM image | EDX spectroscopy mapping | FESEM image | EDX spectroscopy mapping | FESEM image | EDX spectroscopy mapping |
| Black iris / A1 |  (a) |   (b) |  (c) |   (d) | N/A | N/A |
| Clear / A2 |  (e) |   (f) |  (g) |   (h) | N/A | N/A |

Chemical elements descriptions: Fe – iron element, Si – silicon element; N/A – not applicable

Table 3
*FESEM-EDX spectroscopy findings of the brand B: Comparison of chemical elements between Cos-CCL samples with control samples*

| Colour / Sample | Surface analysis | | | | | |
|---|---|---|---|---|---|---|
| | Front | | Back | | Cross-section | |
| | FESEM image | EDX spectroscopy mapping | FESEM image | EDX spectroscopy mapping | FESEM image | EDX spectroscopy mapping |
| Gray iris / B1 |  (i) |   (j) |  (k) |   (l) |  (m) |   (n) |
| Clear / B2 |  (o) |   (p) |  (q) |   (r) | N/A | N/A |

[a]EDX spectroscopy point analysis result – Aluminium element was found in higher count (shown in the highest peak) relative to silicon and oxygen element; Chemical elements descriptions: Al – aluminium element, Si – silicon element; N/A – not applicable

### Chemical elements analysis between Cos-CCL samples of different colours

Table 2 and Table 3 shows the chemical elements of two different colours represented by A1 and B1 samples. Sample A1 contained an iron element as a colour additive and sample B1 contained aluminium element, indicating that chemical elements vary with colours.

## DISCUSSION

In this present study, iron has been found in black iris coloured contact lens whilst grey iris coloured contact lens contained aluminium. We developed an improved protocol by comparing the EDX spectroscopy findings between Cos-CCL samples with their respective clear contact lens control samples and found iron and aluminium elements were only detectable by EDX spectroscopy in the area where the colour additives patterns were visible through FESEM images. For example, the iron element was found on the front surface of the A1 sample in which the colour additives pattern were also imaged by FESEM. In the same sample, silicon was found at the back surface where the colour additive pattern could not be detected by FESEM. Similarly, in the EDX spectroscopy mapping analysis that was performed on B1 sample's front and back surfaces (Cos-CCL sample) the results showed the presence of silicon which was not different from the respective control samples. It was only during the EDX spectroscopy analysis on the separated middle layer of the sample B1 through cross-section image, the aluminium element was detected. Hence, we can conclude that EDX spectroscopy is able to characterise materials on the surface area focused by FESEM.

The characterisation of the chemical elements used as colour additives in Cos-CCL by EDX spectroscopy can be regarded as superior and reasonable compared to the other techniques. In a review article Brennan and Coles (2001) organised the techniques or methods for evaluating contact lens surface into three broad classifications: clinical assessment methods, microscopy and imaging methods and assays method. According to their classification, FESEM-EDX method is classified under the microscopy and imaging category. In order to characterise more complex unknown elements the clinical assessment methods seems unsuitable because it seeks to assess the condition based on Rudko grading system (Rudko & Proby, 1974; Guillon et al., 1992; Hurst et al., 1994) rather than identifying the elements. The assays methods, despite the fact that it can provide promising information on the characterisation of elements, is complicated because it involves a cumbersome sample preparation based on the material being investigated (Wedler, 1977). To date, there is no study conducted to investigate the implication of the chemical elements used as colour additives in Cos-CCL on the human cornea. Thus despite having identifies the presence of iron and aluminium elements in colour additives, their implications on the wearers' eyes cannot be conclusively asserted.

Although this study may provide insights on the biomaterial of Cos-CCL, it has two limitations. First, only one sample for each contact lens was analysed. Second, only a small number of samples, i.e. two different colours of Cos-CCLs were analysed. It can therefore be conclude that users of Cos-CCL should be aware of the chemicals used as colour additives. Even though some chemical elements maybe safe determining whether those 'safe' chemicals have been used is the challenge. Hence, it is preferable to urge manufacturers of cosmetic coloured contact lenses to disclose information regarding the colour additives used in their products.

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