



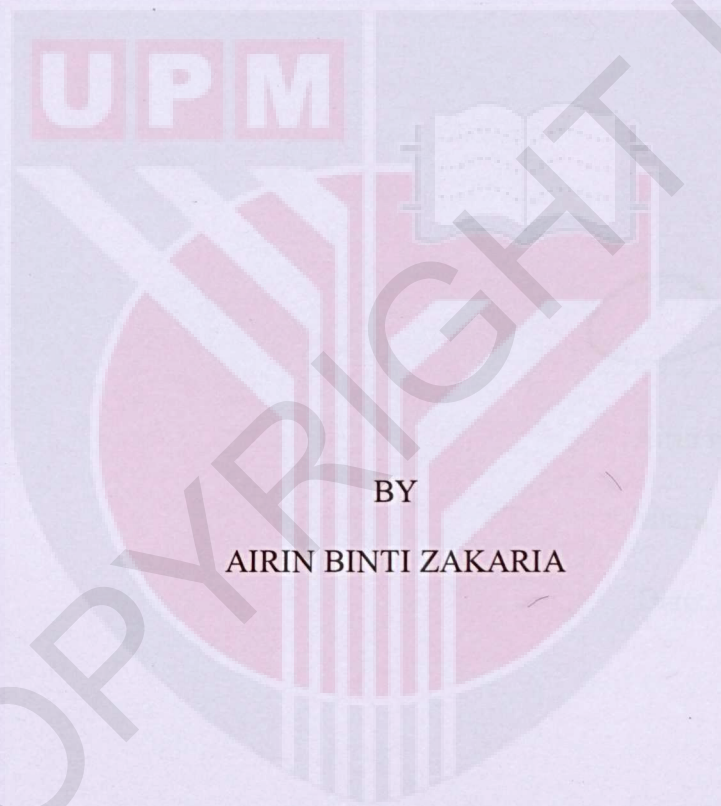
**UNIVERSITI PUTRA MALAYSIA**

***HEAVY METALS IN LIPSTICKS AND ITS ASSOCIATED HEALTH RISK  
TO LIPSTICK CONSUMERS IN UNIVERSITI PUTRA MALAYSIA (UPM)***

**AIRIN BINTI ZAKARIA**

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TO LIPSTICK CONSUMERS IN UNIVERSITI PUTRA MALAYSIA (UPM)**



BY  
AIRIN BINTI ZAKARIA

Thesis submitted in fulfilment of the requirement for the degree of Bachelor Science  
(Environmental and Occupational Health) from the Faculty of Medicine and Health  
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## ABSTRACT

### HEAVY METALS IN LIPSTICKS AND ITS ASSOCIATED HEALTH RISK TO LIPSTICKS CONSUMER IN UNIVERSITI PUTRA MALAYSIA (UPM)

AIRIN BINTI ZAKARIA

**Introduction:** The women are actually exposing themselves towards the heavy metals contained in the lipsticks through ingestion. The lipsticks are reported containing the heavy metals such as lead (Pb), nickel (Ni), aluminium (Al), arsenic (Ar), cadmium (Cd), antimony and chromium (Cr) based on the previous study. Exposure of heavy metals for a prolong time in a small amount might cause a significant probability in developing few suspected chronic health risks and heavy metals associated cancer health risks. The aim of this study is to determine Pb, Cd, Cr concentration in lipsticks of different categories of price in the Malaysian cosmetic market and to evaluate the possibility of developing potential health risk due to daily ingestion of heavy metals in lipsticks among the lipstick consumers. **Methodology:** The questionnaires were distributed to the female staffs in Universiti Putra Malaysia (UPM). The informations obtained were used for the health risk assessment (HRA) to obtain the Hazard Quotient (HQ) and Lifetime Excess Cancer Risk (LCR) via calculation. The microwave digestion technique was chosen for the extraction of heavy metals. The Thermo Scientific iCAP 6000 Series of ICP-OES was used to analyzed the lipsticks samples. **Results:** The concentration of Pb, Cd, and Cr in lipsticks of price category I, II, and III had not exceeded the permissible limit/references dose of IRIS EPA (2013) and Health Canada (2011) except for brown MXX lipsticks in price category III. This lipstick had  $15.440 \pm 22.079$  mg/kg Pb mean concentration which exceed the Canadian Health (2011) limit (10 mg/kg). The concentration of Pb in price category I lipsticks was statistically higher compared to the lipsticks in price category II. The HQ value for all of the lipsticks exposure in every price category was less than 1. The LCR were ranged from  $10^{-9}$  to  $10^{-7}$  for the exposure of Cd in lipsticks while the results for Cr exposure showed that LCR were ranged from  $10^{-5}$  to  $10^{-6}$ . **Conclusion:** The lipsticks sample (N=15) collected from the local markets of Malaysia did contained Pb, Cd, and Cr. Only MXX brown lipsticks contain high Pb concentration when compared to Canada Health (2011) permissible limit. There is low possibility of developing non-carcinogenic and carcinogenic health risk effect due to the exposure on these heavy metals through lipstick consumption.

**Keywords:** Lipsticks, heavy metals, quantitative health risk assessment, ICP-OES, microwave digestion

## ABSTRAK

### LOGAM BERAT DALAM GINCU DAN RISIKO KESIHATANNYA TERHADAP PENGGUNA DI UNIVERSITI PUTRA MALAYSIA ( UPM)

AIRIN BINTI ZAKARIA

**Pengenalan:** Gincu digunakan pada bibir adalah untuk pengguna kelihatan lebih cantik dan menarik, tetapi risiko terhadap wanita-wanita ini adalah pendedahan diri mereka kepada logam berat yang terkandung dalam gincu. Gincu dipercayai mengandungi logam berat seperti plumbum, nikel, aluminium, arsenik, kadmium, antimony, dan kromium berdasarkan kajian sebelumnya. Pendedahan terhadap logam berat untuk masa yang berpanjangan walaupun ia adalah dalam jumlah yang kecil berkemungkinan akan menyebabkan kan risiko kesihatan kronik dan juga kanser yang dikaitkan dengan logam berat akibat daripada pendedahan yang berlaku. Tujuan utama kajian ini adalah untuk menentukan kepekatan logam berat dalam gincu kategori harga yang berlainan di pasaran Malaysia dan menilai kemungkinan potensi risiko kesihatan yang mungkin terjadi disebabkan pendedahan harian terhadap logam berat dalam gincu untuk wanita yang memakai gincu di bibir mereka. **Metodologi:** Soal selidik diedarkan kepada staf wanita Universiti Putra Malaysia ( UPM) bagi mendapatkan maklumat yang berkaitan dengan jenama dan harga gincu yang biasa mereka gunakan dan maklumat sosio demografi responden yang akan digunakan untuk kesihatan penilaian risiko “Hazard Quotient” (HQ) dan pengiraan terhadap “Lifetime Excess Cancer Risk” (LCR). Teknik penghadaman Microwave dipilih untuk mengenalpasti kandungan logam berat kerana mempunyai keupayaan dan kecekapan mengenal pasti plumbum yang tinggi. TermoSaintifik ICAP 6000 Siri (ICP-OES) telah digunakan untuk menganalisis sampel gincu itu. ICAP 6000 Siri menggunakan perisian iTEVA untuk menjalankan analisis. **Hasil:** Pb min kepekatan tinggi dalam warna coklat / semulajadi gincu berbanding dengan gincu merah dan merah jambu. Terdapat perbezaan yang signifikan terhadap kepekatan Pb Antara kategori harga gincu I dan II. Secara amnya, HQ dan LCR menunjukkan risiko kesihatan yang boleh diterima dan risiko kesihatan dengan jelas boleh diterima kerana pendedahan terhadap Pb, Cd, dan Cr dalam sampel gincu itu. **Kesimpulan:** Kepekatan min Pb, Cd, dan Cr dalam sampel gincu (n = 15) dipasaran tempatan Malaysia telah diukur. Tahap logam berat dalam gincu yang dianalisis tidak melebihi had yang ditetapkan dalam Health Canada (2011) dan IRIS, EPA (2013).

**Kata Kunci:** gincu, logamberat, penilaian risiko kesihatan, ICP-OES, penghadaman gelombang

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## LIST OF ABBREVIATIONS

Pb	Lead
Cd	Cadmium
Cr	Chromium
ADD	Average Daily Dose
LADD	Lifetime Average Daily Dose
HQ	Hazard Quotient
LCR	Lifetime Excess Cancer Risk
RfD	Reference Dose
CSF	Cancer Slope Factor
IRIS	Intergrated Risk Information System
EPA	Environmental Protection Agency
SD	Standard deviation
SPSS	Statistical Package for Social Science

## CHAPTER 1

### 1. INTRODUCTION

#### 1.1 Background

Metal content in lip products has been a health issue of concern internationally. It is because the lipsticks is the daily basis products that are included in the face make up application other than the face powder, foundations, eye shadows and blusher (Piccinini *et al.*, 2013). Naturally the lipstick is applied on the lips for the users to look more beautiful and attractive, but the price for these women to be beautiful is by exposing themselves towards the heavy metals contained in the lipsticks. The lipsticks are believed to contain the heavy metals such as lead, nickel, aluminium, arsenic, cadmium, antimony and chromium based on the previous study (Al-Saleh and Al-Enazi, 2011). Some of the function of this heavy metal such as lead is basically to provides the pigment in colored lipsticks (Gondal *et al.*, 2010). Results of Piccinini *et al.* (2013) study showed that 48% of all tested purple lipsticks sample contained more than 1 mg/kg of lead, whereas this proportion was lower for the other colours (33% for brown, 30% for pink, and 14% for red) even though statistically the average lead content for the various combinations of shades did not show any significant difference at 95% probability.

The possible influences of the price on lead level was also evaluated for both lip product types separately considering the important difference in lead content within the lipsticks. On the contrary, in the case of lipsticks which presented 31% of items containing more than 1 mg/kg of lead (46 out of 149), the samples belonging to expensive price category statistically showed the highest quality (lower lead content), followed by products in price categories intermediate and lower (Piccinini *et al.*, 2013).

The exposure of heavy metals to the lipsticks consumers are only in small amount but they are exposing towards it for a prolong time which make it significant in developing few suspected chronic health risk and also the cancer. The application of lipsticks on the lips might expose to a minute amount of the lipsticks consumer through the ingestion route which can occur when the consumer are eating food and licking on the lips. Chronic health risk is an irreversible response characterized by a gradual onset of long duration, following a constant or continuous exposure period to a low toxicant dose. The toxicant doses that will be studied in this research are the level or dose of heavy metals in the lipsticks.

The chronic health risk that might be develop from exposure towards Lead (Pb) is the neurological effects, teratogenic effects, and blood systemic effects (Gondal *et al.*, 2010). The chronic health risks of exposing towards Chromium (Cr) are the irritation to the nose, runny nose, nosebleeds, and ulcers and perforations in the nasal septum cause by the breathing of high levels of chromium. Ingesting large

amounts of Cr can cause stomach upsets and ulcers, convulsions, kidney and liver damage, and even death. Skin contact with certain Cr compounds can cause skin ulcers. For people who are extremely sensitive to Cr, allergic reactions consisting of severe redness and swelling of the skin have been noted (Gondal *et al.*, 2010). The chronic health risk of the Nickel (Ni) is reproductive disorders and effects on foetuses (Chashschin *et al.*, 1994).

The prolong exposure of Cadmium (Cd) towards human are closely linked with cardiovascular diseases, such as atherosclerosis and hypertension (Angeli *et al.*, 2013). While for the Arsenic (As) health risk, long term As exposure damages several internal organs and has been linked to bladder, lung, and skin cancers (McClintock *et al.*, 2012). The Aluminium (Al) may cause neurotoxic effects for the possible prolong exposure of the lipsticks consumer towards this heavy metals (Nieboer *et al.*, 1995).

Other than the non-carcinogenic chronic health risk, the exposures towards heavy metals might also caused carconigenic health risk to the lipstick consumers. The cadmium pigments that were to be considered carcinogenic, but they are less potent as carcinogens. These arguments are based upon the opinion that the carcinogenicity of cadmium sulfide (CdS) pigment as administered in the chronic rat inhalation (US Department of Labor, 2010).The cadmium pigment is less potent because of the Cd compound characteristics which is less soluble and hence less bioavailable in human tissue.

For an effective determination of heavy metals concentrations in lipsticks sample, reference on few literature was carried out to find out the efficient lipstick digestion procedure and heavy metals determination technique. Several reports and research papers were published on the presence of trace levels of heavy metals in both cosmetic and food products and multiple methods of sample preparation and analysis have been proposed (Piccinini *et al.*, 2013). The amount of Pb extracted depends significantly upon experimental conditions such as sample weight, combination of used acids, temperature and decomposition procedure. In view of sample decomposition various methods are available: the dry ash one and sample digestion by means of a combination of diverse acid mixtures both in an open system and a microwave digester (Al-Saleh and Al-Enazi, 2011). Microwave digestion is one of the digestion technique needed in sample preparation for the heavy metals analysis in the samples. The raw samples are weighed between 0.3-0.5 g weight range before inserted into the vessel to be digested using the microwave instruments to get the final clear solution. The most common analytical techniques applied to quantify lead in cosmetics and food products include: Inductively Coupled Plasma-Mass Spectrometry (ICP-MS), and Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) (Hepp, 2012). The clear solution obtained from the microwave digestion will be used for the sample analysis using instrument stated above. The results obtained from the sample analysis are required for the evaluation of the daily intake of heavy metals among consumers and assessed the health risk due to consumption of the lipsticks sample.

The evaluations of potential health risk with respect to the daily consumption of lipsticks for adults women are based on some insight of heavy metal content in the lipsticks and serve as a basis for comparison to the Standard of Health Canada and IRIS US EPA. The main aim of this study is to determine the heavy metals concentration in lipsticks of a different range of price in the Malaysia market and evaluate the possibility of potential health risk due to daily ingestion of heavy metals in lipsticks to the women who applied lipsticks on their lips.

## 1.2 Research Justification

There are millions of cosmetics consumer have use lipsticks as their daily basis. Although the direct contact exposure rarely results in the penetration of lipsticks substance through the skin and cause the systemic exposure, it still can produce exposure along oral cavity since it is applied on the lips. In Malaysia, there is still no published research study involving the concentration of heavy metals such as lead, chromium, nickel, and arsenic in the lipsticks that have been selling in the market. The health risks towards the cosmetics consumers that associated to the use of the lipsticks containing heavy metals have not been studied yet. It is a necessity for these problems to be further studied so that there will be baseline information on this problems and it can be a reference for the future study on heavy metals in lipsticks in Malaysia.

The top popular lipsticks used by the staff of UPM are going to be use as sample for the entire study. There is study proved that lead content in red lip products was statistically higher than that in other colors such as red and pink lip products. Other than that, the higher price of lip products is correlated with a higher quality in terms of lower lead concentration. The lower average lead content was measured in lipsticks belonging to high price category compared to a low market price of the samples (Piccinini *et al.*, 2013). This previous study showed that the price does correlate to the concentration level of heavy metals in lipsticks. This is the reason for the sample to be analyzed based on its price range. The study of Wang *et al.* (2010) had analyzed ten lipsticks collected in Chengdu market in order to determine the concentration lead in the lipsticks. In a study of Al-Saleh and Al-Enazi (2011), the samples are taken from total of 28 lipsticks from 14 brands widely sold in the local Saudi Arabian market were purchased from cosmetic shops.

The survey for the commonly lipstick usage will be done on women who either work as a lecturers, staffs or as students of Universiti Putra Malaysia (UPM). This is because working women usually applying lipsticks on the working day. Some students are also applying lipsticks when they are going to the class to increase their self-confidence. The reason for the respondents to be the lecturers, staff, and the students because in the study of Biesterbos *et al.* (2013), more users with a high level of education used the care products compared to users with an intermediate or low level of education.

The health risk assessment due to the exposure towards heavy metals in lipsticks are using Hazard Quotient (HQ) for the non-carcinogenic chronic health effects and Lifetime Excess Cancer Risk (LCR) for the carcinogenic chronic health effects. Both formula are calculated by combining the data of the heavy metals mean concentration in the lipsticks sample and sociodemographic data of the respondents (NITE, 2011).

### 1.3 Problem Statements

The study of Piccinini *et al.* (2013) revealed that 49 samples (22%) contained lead at a level higher than 1 mg/kg which is done in European country. To date, there is no study reported on the occurrence of heavy metals in lipsticks sold in Malaysia. The results of the Piccinini *et al.* (2013) study also appears that heavy metals concentration in the lip products sold in the European market is below all the limits of German Federal Government, Health Canada, and FDA standards. This study is important to determine the baseline information on either the heavy metals content of lip products available on the Malaysian market is well below all the listed standards limits. It has been recently reported by the US Food and Drug Administration (FDA) and European Union's Restriction on Hazardous Substances (ROHS) directive that some cosmetic materials used by humans contain hazardous substances which is the heavy metals that has been highlighted in this study (Gondal *et al.*, 2010). Due to these reasons, there is a growing concern that some of the cosmetic products used by

humans contain hazardous substances which are injurious to health. It has gained attention over the past years, since they are a possible source of daily, population-wide and often long-term exposure to a variety of chemicals. To be precise, there are still less research that study on the potential health risk that may develop due to the prolong exposure of this heavy metals content of lipsticks towards the consumer here in Malaysia. This study is a necessity to determine if there are significant association between the exposures of certain amount of heavy metals in lipsticks may cause any potential health risk towards the lipsticks consumer.



## 1.4 Conceptual Framework

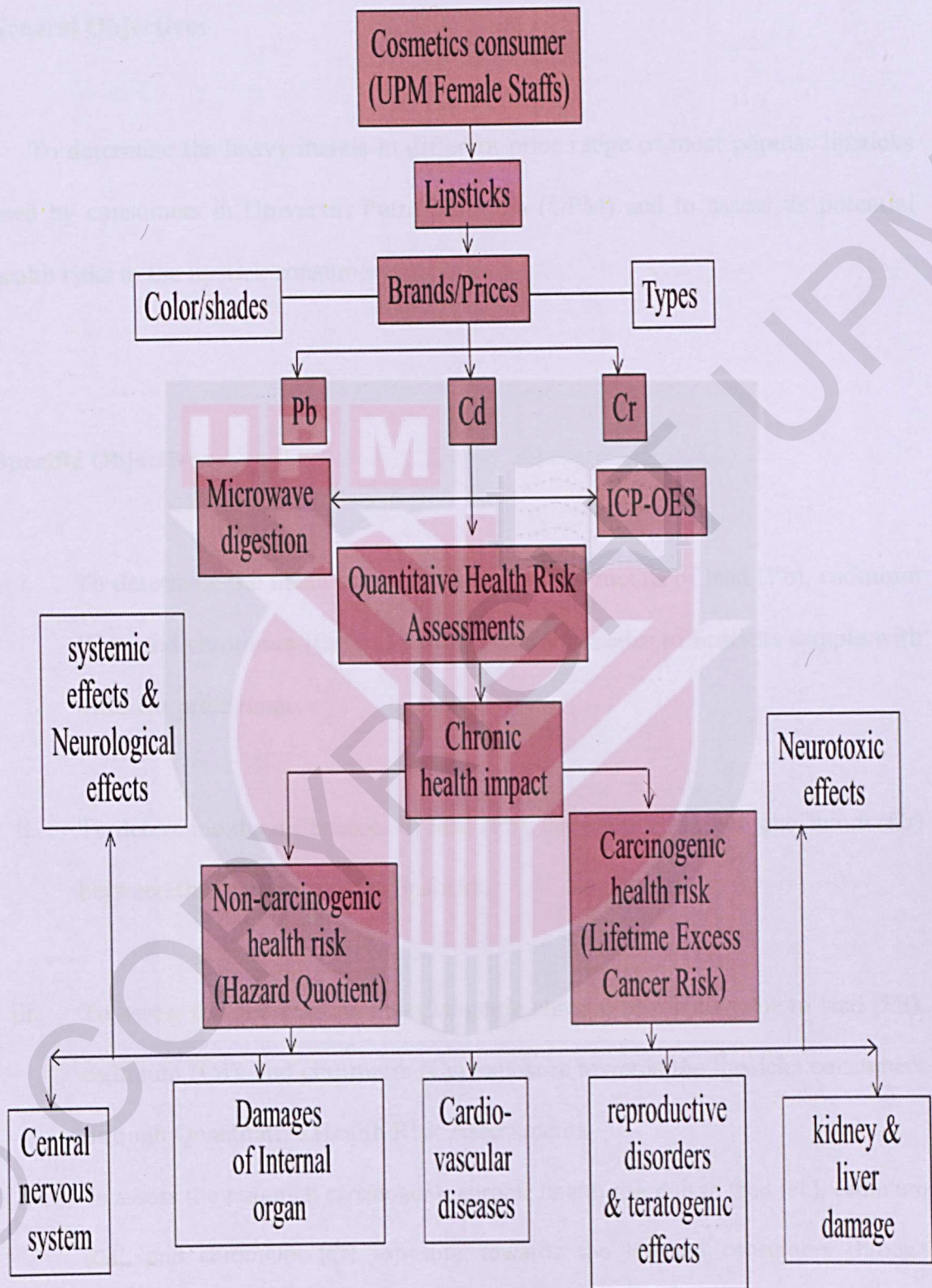


Figure 1.1. Conceptual framework of the study

## 1.5 Objectives

### General Objectives

To determine the heavy metals in different price range of most popular lipsticks used by consumers in Universiti Putra Malaysia (UPM) and to assess its potential health risks to the lipstick consumers.

### Specific Objectives

- i. To determine the mean concentration of heavy metals of lead (Pb), cadmium (Cd), and chromium (Cr) in 15 different color shades of lipsticks sample with different price range.
- ii. To determine the differences of lead (Pb), cadmium (Cd), and chromium (Cr) between the price categories lipsticks.
- iii. To assess the potential non-carcinogenic chronic health risk due to lead (Pb), cadmium (Cd), and chromium (Cr) exposure towards the lipsticks consumers through Quantitative Health Risk Assessments.
- iv. To assess the potential carcinogenic chronic health risk due to lead (Pb), cadmium (Cd), and chromium (Cr) exposure towards the lipsticks consumers through Quantitative Health Risk Assessments.

## 1.6 Hypothesis

- i. There are the concentrations of lead (Pb), cadmium (Cd), and chromium (Cr) in 15 different color shades of lipsticks sample with different price range.
  - ii. The price of the lipsticks is significantly correlated to the occurrence of lead (Pb), cadmium (Cd), and chromium (Cr) in the lipsticks.
  - iii. The lead (Pb), cadmium (Cd), and chromium (Cr) is significantly different between the low, intermediate, and high price categories of the lipsticks.
  - iv. The Hazard Quotients (HQ) is significantly showing possibility of developing non-carcinogenic chronic health risk due to the exposure of lead (Pb), cadmium (Cd), and chromium (Cr) in lipsticks.
  - v. The Lifetime Cancer Risk (LCR) is significantly showing possibility of developing carcinogenic chronic health risk due to the exposure of lead (Pb), cadmium (Cd), and chromium (Cr) in lipsticks.
- CHAPTER 2

## CHAPTER 2

### 2. LITERATURE REVIEW

#### 2.1 Current Cosmetics Users In The World

Personal care products (PCP) are widely and regularly used by people on a daily basis (Wu *et al.*, 2010). The cosmetics are part of these PCP. In recent study, there are results that the PCP consumers have combined use of several PCPs. There are high percentage of respondents (70–95%) used a combination of two skin care products, for example, day cream and night cream (Biesterbos *et al.*, 2013). The Wu *et al.* (2010) study showed results with high significant that the PCP consumer who used of shampoo will also use the hair conditioner which increase the exposure of chemicals content in the PCP including the heavy metals. In the study of Biesterbos *et al.* (2013), the percentages of female users were higher ( $p < 0.05$ ) compared to the percentages of male users for all PCPs studied, except for shaving products. The percentage of users in the age group 18–39 (young) was higher compared to the percentages of users in the older age groups for most products. The hair dye was most often used by middle aged and low intermediately educated respondents. In addition, the level of education influenced the use of several other PCPs. More users with a high level of education used aftershave compared to users with an intermediate or low level of education.

Based on the previous study, it is concluded that the cosmetics consumers are among the women in young age group with a high level education which is compatible with the target group of female staffs of Universiti Putra Malaysia.

## 2.2 Lipsticks Types And Usage

Since the prehistoric times, natural based lipsticks (colorful fruit and plant juices) were used by women. “Kiss of Death” is a term that is commonly associated with lipstick due to some ancient civilizations unknowingly used poisonous materials for making of lipstick.

Lipsticks can be separated in several types – ordinary lipstick, glossy or glittery lipsticks, chapstick, moisturizing lipsticks, lip gloss and lip balm (Lipsticks History, 2010).

### Moisturizing lipsticks

Moisturizing lipstick will keeps lips soft and smooth so people who have dry lips are suitable to use it. These lipsticks moisturize lips because of ingredients like vitamin E, glycerin and aloe. Other benefits of using moisturizing lipsticks are wet and very shiny lips (Lipsticks History, 2010).

## Satin and Sheer lipsticks

These lipsticks also moisturize and nourish lips and make it shiny and glossy and also the other choices for dry lips are satin and sheer lipsticks.. Sheer and satin lipsticks have high oil ingredients and they can appear darker in the package than they are on lips. Another characteristic of lipsticks with oil components is that it should be reapplied many times (Lipsticks History, 2010).

## Matte lipstick

Mate lipsticks are perfect choice for women who are looking for colorful and nice shade. These lipsticks have effect of flat and not shiny lips. Your lips can look smother and younger with matte lipstick. Also it is recommended to combine products with vitamin E and aloe with mate lipsticks (Lipsticks History, 2010).



Figure 2.1. Matte Lipsticks

### Cream lipsticks

Cream lipsticks are suitable for the women with small lips. Lipstick with cream formula is not so shiny, but it has smooth effect on lips. You can use lip gloss afterward for desire look. Cream lipsticks contain more wax in order to protect lips, but also can cause effect of dry lips (Lipsticks History, 2010).

### Pearl and Frosted lipstick

Frosted lipstick makes lips sparkle and glisten. Pearl and frosted lipstick reflects light and makes very shiny effect on your lips. Negative effects are that can cause lips feel heavy, crack and dry. It is recommended to moisturize your lips before use this lipsticks (Lipsticks History, 2010).

### Gloss Lipstick

Gloss is very popular lipstick for women with thin and small lips because make lips shine and enhance the dimension of depth. Gloss can be combined with traditional lipstick (Lipsticks History, 2010).



Figure 2.2. Gloss Lipstick

#### Long wearing and transfer-resistant lipsticks

Women who don't have time to apply lipstick frequently may use long wearing lipsticks. These lipsticks have formula that keep lips look perfect from 4 to 8 (or more). They are long lasting until you eat something greasy or oily. Some of them contain moisturizer to balance the dryness of lips (Lipsticks History, 2010).

### 2.3 Heavy Metals in Lipstick Products and Its Legal Requirements

The results of Al-Saleh and Al-Enazi (2011) proved that amounts of heavy metals of lead, cadmium, nickel, chromium, mercury, antimony, and arsenic were found in the 28 samples of lipsticks. In this study, the heavy metals that will be

studied are the lead, chromium, and cadmium. There are no specific regulations on heavy metals concentrations in the cosmetic products here in Malaysia.

However, there are regulations on controlling the cosmetic products in the Malaysia cosmetics market which is under the Control of Drugs and Cosmetic Regulations 1984. The control of cosmetic products in Malaysia was through pre-market registration; in which the company is responsible for placing the product in the local market by submitting the product details and relevant certificates for review and registration by the National Pharmaceutical Control Bureau (NPCB) before the product can be manufactured, imported or sold in the country from year 2002 to the end of year 2007 (NCPB, 2014).

This system has been replaced by a notification procedure starting 1 January 2008 to harmonize with cosmetic regulations in the ASEAN region. The companies now are required to only notify / declare their compliance to the ASEAN Cosmetic Directive to the NPCB. Post Market Surveillance will be actively conducted by the NPCB to ensure compliance. Action will be taken against any product that is not compliant with the Directive. The Guidelines for Control of Cosmetic Products in Malaysia under the Control of Drugs and Cosmetic Regulations 1984 is an adaptation to the requirements of the ASEAN Cosmetic Directive (NCPB, 2014).

In this regulation, it is the responsibility of the company that bring in the products into the local market to ensure that the cosmetics are complying the

regulations. It is included the requirements of the cosmetics for not containing elements listed in Annex I, II, and II including the heavy metals. Cosmetic products must not contain any substance that may cause harm to the consumer when applied under normal or reasonably foreseeable conditions of use. In Annex II, the heavy metals included lead, chromium, and cadmium is listed as the substances which must not form part of the composition of cosmetic products (NPBC, 2014).

While in Europe, there is European legislation on the approximation of the laws of Member States related to cosmetic products was introduced in 1976 by the EU Directive 76/768/EEC, known as the Cosmetic Products Directive, which has been frequently amended. The new Cosmetic Products Regulation (EC Reg. 1233/2009) was adopted in November 2009 to replace the Directive 76/768/EEC; which most of its provisions will be only applicable from the 11th July 2013. Lead is listed in Annex II of both Directive 76/768/EEC and EC Reg. 1233/2009, among the substances which must not form part of the composition of cosmetic products (Piccinini et al., 2013).

However, as stated in article 17 of EC Reg. 1233/2009, “the non-intended presence of a small quantity of a prohibited substance, stemming from impurities of natural or synthetic ingredients, the manufacturing process, storage, migration from packaging, which is technically unavoidable in good manufacturing practice, shall be permitted provided that such presence is in conformity with article 3”, which in turn requests the cosmetic products available on the market to be safe for human health

when used under normal or reasonably foreseeable conditions of use (Piccinini et al., 2013).

A limit for lead in cosmetics has been defined neither at European nor at international level. The occurrence of lead in cosmetic samples can originate from some ingredients naturally containing lead (such as dyes and pigments) and/or from being introduced during the production process (Piccinini *et al.*, 2013).

Unfortunately there are no current international standards for impurities like heavy metals in cosmetics except 20 µg/g for lead and 5 µg/g for cadmium (Al-Dayl *et al.*, 2011). The Canadian regularity limits for certain metals in cosmetics are 10 µg/g for Pb, 3 µg/g for As, Cd, Hg and 5 µg/g for Sb (Health Canada, 2011). These limits are based to provide a high level of protection to susceptible consumer (Al-Dayl *et al.*, 2011).

In 2010, the International Cooperation on Cosmetics Regulation (ICCR) set up a Working Group on Traces in Cosmetics which aimed to provide the highest level of global consumer protection, while minimising barriers to international trade. ICCR is an international group of cosmetic regulatory authorities from the United States (Food and Drug Administration), Japan (Ministry of Health, Labour and Welfare), the European Union (European Commission, Directorate General Enterprise) and Canada (Health Canada). In the frame of the activities of the Working Group on Traces, the issue of the possible presence of lead in cosmetics has

been taken into consideration with the intention of drafting a recommendation for lead levels in cosmetic products. In this view, the awareness of what is currently available on the market is key data that could provide information on what can be technically achievable and on the levels of lead to which the consumers are exposed. The lip items were considered interesting due to the fact that a significant part of applied product is likely to be ingested so that exposure is not only based on dermal contact.

Other than that, knowledge about the prolong use of cosmetics products and its subsequent health effects is lacking. A recent survey conducted in 2004 showed most of the US girls aged 7-19 with 63 percent are using lipsticks (Ullah *et al.*, 2013). We ingest the ingredient of lipsticks when the lips are licked, eat and drink, kisses someone while wearing lipsticks. The finding showed that women are exposed in to heavy metals in lipsticks as early as 7 years.

Therefore, there is a need for the establishment of regulations for heavy metals in personal care products as this will act to prevent harm in advance from the use of these products especially the lipstick. The assessment of human risk from the exposure to cosmetics which are highly contaminated with heavy metals need to be done (Ullah *et al.*, 2013). The ubiquity of cosmetics and the lack of their regulations encouraged us to test a sample of lipsticks them for various trace metals such as lead, cadmium, nickel, chromium, mercury, antimony, and arsenic since the EU Cosmetics

Directive lists these metals in the category of compounds (Annex II) that are prohibited for use in cosmetic products (Al-Saleh and Al-Enazi, 2011).

Table 2.1. Results of heavy metals analysis in few lipsticks

Sample	Brand	Parent company	Lipstick line Shade	Lot #	Lead (Pb) (ppm)
1	Maybelline	L'Oréal USA	Color Sensational 125 Pink Petal	FF205	7.19
2	L'Oréal	L'Oréal USA	Colour Riche 410 Volcanic	FE259	7.00
3	NARS	Shiseido	Semi-Matte 1005 Red Lizard	0KAW	4.93
4	Cover Girl Queen Collection	Procter & Gamble	Vibrant Hues Color Q580 Ruby Remix	9139	4.92
5	NARS	Shiseido	Semi-Matte 1009 Funny Face	9DLW	4.89
6	L'Oréal	L'Oréal USA	Colour Riche 165 Tickled Pink	FF224	4.45
7	L'Oréal	L'Oréal USA	Intensely Moisturizing Lipcolor 748 Heroic	FD306	4.41
8	Cover Girl	Procter & Gamble	Continuous Color 025 Warm Brick	9098	4.28
9	Maybelline	L'Oréal USA	Color Sensational 475 Mauve Me	FF201	4.23
10	Stargazer	Stargazer	Lipstick 103 c	180808	4.12

Sources: FDA (2010)

## 2.4 Lead (Pb)

Lead affects virtually every system in the body such as the reproductive, neurological, hematopoietic, hepatic, and renal systems (Al-Saleh *et al.*, 2009). It is widely known to be toxic to the reproductive system, the liver and kidney, and the immune system but evidence for lead as a carcinogen is still considered less clear (Silbergeld *et al.*, 2000).

The lead poisoning may lead to chronic health risk such as poor muscle coordination, nerve damage to the sense organs and nerves controlling the body, increased blood pressure, hearing and vision impairment, reproductive problems (e.g., decreased sperm count), and retarded fetal development even at relatively low exposure levels if exposed to the pregnant women among adults (USEPA, 2013).

## 2.5 Cadmium (Cd)

Cadmium is a metal with high toxic effects. It has an elimination half-life of 10–30 years, and accumulates in the human body, particularly the kidney (Nawrot *et al.*, 2006). Cd exposure through Chronic inhalation and oral exposure of humans can result in a build-up of cadmium in the kidneys that can cause kidney disease, including proteinuria (USEPA, 2013). Cd has been classified as a probable human carcinogen (Group B1) (USEPA, 2013). The example of the carcinogenic health risk

that may develop due to Cd exposure through oral exposure is breast cancer based on the study of (Nawrot *et al.*, 2006).

## 2.6 Chromium (Cr)

The oral Reference Dose (RfD) of Cr is 3 mg/kg/day based on the assumption that thresholds exist for certain toxic effects such as cellular necrosis. In general, the RfD is an estimate (with uncertainty spanning perhaps an order of magnitude) of a daily exposure to the human population (including sensitive subgroups) that is likely to be without an appreciable risk of deleterious effects during a lifetime. The Cr RfDs can also be derived for the noncarcinogenic health effects of substances that are also carcinogens. Therefore, it is essential to refer to other sources of information concerning the carcinogenicity of this substance. If the U.S. EPA has evaluated this substance for potential human carcinogenicity, a summary of that evaluation will be contained in Section II of this file.

In 1965, a study of 155 subjects exposed to drinking water at concentrations of approximately 20 mg/L was conducted outside Jinzhou, China. Subjects were observed to have sores in the mouth, diarrhea, stomachache, indigestion, vomiting, elevated white blood cell counts with respect to controls, and a higher per capita rate of cancers, including lung cancer and stomach cancer. Precise exposure concentrations, exposure durations, and confounding factors were not discussed, and

this study does not provide a NOAEL for the observed effects. However, the study suggests that gastrointestinal effects may occur in humans following exposures to hexavalent chromium at levels of 20 ppm in drinking water (USEPA, 2012).

Dermal exposure to chromium has been demonstrated to produce irritant and allergic contact dermatitis (Bruynzeel *et al.*, 1988; Polak, 1983; Cronin, 1980; Hunter, 1974). Primary irritant dermatitis is related to the direct cytotoxic properties of chromium, while allergic contact dermatitis is an inflammatory response mediated by the immune system. Allergic contact dermatitis is a cell-mediated immune response that occurs in a two-step process. In the first step (induction), chromium is absorbed into the skin and triggers an immune response (sensitization). Sensitized individuals will exhibit an allergic dermatitis response when exposed to chromium above a threshold level (Polak, 1983). Induction is generally considered to be irreversible. Concentrations of hexavalent chromium in environmental media that are protective of carcinogenic and noncarcinogenic effects are likely to be lower than the concentrations required to cause induction of allergic contact dermatitis. However, these concentrations may not be lower than concentrations required to elicit an allergic response in individuals who have been induced (USEPA, 2012).

## **2.7 Microwave Acid Digestion**

The study of (Wang *et al.*, 2010) had taken lipsticks sample 0.1g and added it into microwave digestion tank. Then nitric acid - hydrogen peroxide mixture was

added. Then microwave digestion began. After digestion and cooling, digestion fluid were removed into flask with 1% nitric acid solution washing several times, combined in a flask in 25mL, with 1% nitric acid constant volume before analyzing it for heavy metals concentration by using ICP-MS.

The method of Hepp (2012) for the digestion procedure was as follows: 0.2 g of each sample was weighed into a microwave vessel liner, subsequently 7 mL of nitric acid (69%) and 2 mL of hydrofluoric acid (47–51%) were added. The liners were placed in vessels, closed with a sealed cap and put into the microwave oven. After the second step of the programme, vessels were cooled to 50 ° C and vented, then 30 mL of 4% boric acid aqueous solution was added to each sample and the vessels were further heated in the microwave, in order to complex and dissolve any insoluble fluorides (step 3). Each sample has been analysed at least in duplicate, together with two method blanks for each digestion batch. The digestion sample were then cooling and venting.

For the samples processed, a method blank was carried throughout the entire sample preparation and analytical process. These blanks will be useful in determining if samples are being contaminated (US EPA, 1996).

The high recovery values obtained from the six replicates of the reference materials that were analysed confirmed the method extraction efficiency (Piccinini *et al.*, 2013)

## 2.8 Inductively Coupled Plasma Optical Emission Spectrometry (ICP-OES)

The general understanding to operate the ICP-OES is important in order to generate a high quality data. The aqueous sample is converted to aerosols via a nebulizer is the basic principle of ICP-OES. The mechanism of the ICP-OES is that the aerosols are transported to the inductively coupled plasma which is a high temperature zone (8,000–10,000°C). The characteristic of optical emissions (lights) are produced when the analytes are heated (excited) to different (atomic and/or ionic) states. These emissions are separated based on their respective wavelengths and their intensities are measured (spectrometry). The intensities are proportional to the concentrations of analytes in the aqueous sample. The quantification is an external multipoint linear standardization by comparing the emission intensity of an unknown sample with that of a standard sample. Multi-element calibration standard solutions can be prepared from single- and multielement primary standard solutions. With respect to other kinds of analysis where chemical speciation is relevant (such as the concentration of ferrous iron or ferric iron), only total elemental concentration is analyzed by ICP-OES (Madison, 2005).

The brief and general procedure in operating ICP-OES is five working standard solutions are prepared from independent primary standard solutions. The newly prepared working standard solutions are confirmed against old working standard solutions and against other independent primary standard solutions. In daily operation, the ICP-OES instrument is started brought to operation conditions and let

stabilized. The sample introduction system is checked and the wavelengths are tuned. The instrument is standardized with the five working standard solutions (multi-point linear fitting). Samples are measured with standardization blanks, other kinds of blanks, drift control samples, and quality control samples. After batches of samples are measured, the data are downloaded to an Excel spreadsheet. The data are corrected in terms of standardization blanks, other relevant (Madison, 2005).

## 2.9 Quantitative Health Risk Assessment

The potential chronic health risk due to the exposure of heavy metals to the cosmetics consumers can be divided into two; the non-carcinogenic and carcinogenic risk. The non-carcinogenic risk is expressed using the hazard quotient (HQ). For the ingestion exposure of the specific toxicant (heavy metals), HQ is the ratio of the average daily dose (ADD) to the reference dose (RfD). It compares the magnitude of ADD (Estimated Average Daily Dose) with that of RfD (Reference Dose). If HQ is larger than 1, i.e., if ADD exceeds RfD, there is risk. If HQ is smaller than 1, i.e., if ADD does not exceed RfD (NITE, 2011). For carcinogenic health risk, the toxicant body burden is indicated by the Lifetime Cancer Risk (LCR). LCR is the product of Lifetime Average Daily Dose (LADD) and the Cancer Slope Factor (CSF). The risk to develop cancer due to the lifetime exposure towards the heavy metals in lipsticks is acceptable if the LCR value is between  $10^{-4}$  –  $10^{-6}$  (NITE, 2011).

## CHAPTER 3

### 3. METHODOLOGY

#### 3.1 Study Design

This study was the cross-sectional study. The lipstick samples were purposively taken based on the survey among the respondents; the female staffs of lipsticks consumer in the Universiti Putra Malaysia on the brand and price of the lipsticks that they usually applied. The target samples were the most popular lipsticks among the respondents (N=215). 15 lipsticks of 10 different lipsticks brands were collected over 27 lipsticks brands used by the respondents.

In this study, the lipsticks samples (N=15) were collected by purchasing the lipsticks at cosmetics shop and stalls. The lipsticks samples were categories according to their price; “cheap” (category I <RM29.99) (n=5), “intermediate” (category II- RM30-RM59.99) (n=5), and “expensive” (category III >RM60) (n=5). The lipsticks samples (n=15) were chosen based on the 5 top popular lipsticks used by the respondents in each of the price categories as stated in Figure 4.1, 4.2, and 4.3. The colors of the lipsticks in different price categories were pink (n=11), red (n=2) and nude/natural-like color (n=2). The brands of the lipsticks were totally different according to the price categories: “cheap” (category I -red Sxxxx Gxxx, pink Sxxxx Gxxx, brown Sxxxx Gxxx, pink Mxxxxxxxx, and pink Axxx, “intermediate”

(category II- pink Sxxxxx Sxxx, pink Lxxxxx Pxxxx, pink Bxxxxxxx, red Rxxxxx, and pink Rxxxxx), and “expensive” (category III- pink Exxxx Lxxxxx, pink Mxx, red Mxx, brown Mxx, and pink Cxxxxx).

The 15 lipstick samples were analyzed in triplicates that make it in total of 45 samples in this study. Despite a small sample size reported in this study, previous study also reported the concentration of lead was analyzed in ten lipsticks collected in Chengdu market (Wang *et al.*, 2010). The study of (Al-Saleh and Al-Enazi, 2011) had also reported a small sample size where 28 of the lipsticks collected from the brands widely sold in the local Saudi Arabian to be analysed for heavy metals concentration.

### **3.2 Study Instruments**

#### **3.2.1 Questionnaires**

The questionnaires were distributed to the female staffs of Universiti Putra Malaysia (UPM) in order to obtain the information related to the brand and price of the lipsticks that they are usually applied. The questionnaire distributed based on the list of female staffs in UPM that had been obtained through Human Resources Office of UPM administration.

The duration and frequency of applying the lipsticks were included in the questionnaire. The details of sociodemographic information of the female staff were also obtained from the questionnaire such as the weight of the respondent, the frequency of applying the lipsticks in a day, and the duration of the respondent use the lipsticks in her whole life. These information were used for the health risk assessment of Hazard Quotient (HQ) and Lifetime Excess Cancer Risk (LCR) calculation.

There are 3201 female staffs working in UPM based on the list obtained from the Human Resources Office of UPM administration, the total sample size required in this study were 374 respondents according to the sample size calculation. The formula of sampling is calculated as below:

$$N = \frac{t^2 \times p(1-p)}{m^2} \quad (\text{Eq.3.1})$$

**where,**

**n** = required sample size

**t** = confidence level at 95% (standard value of 1.96)

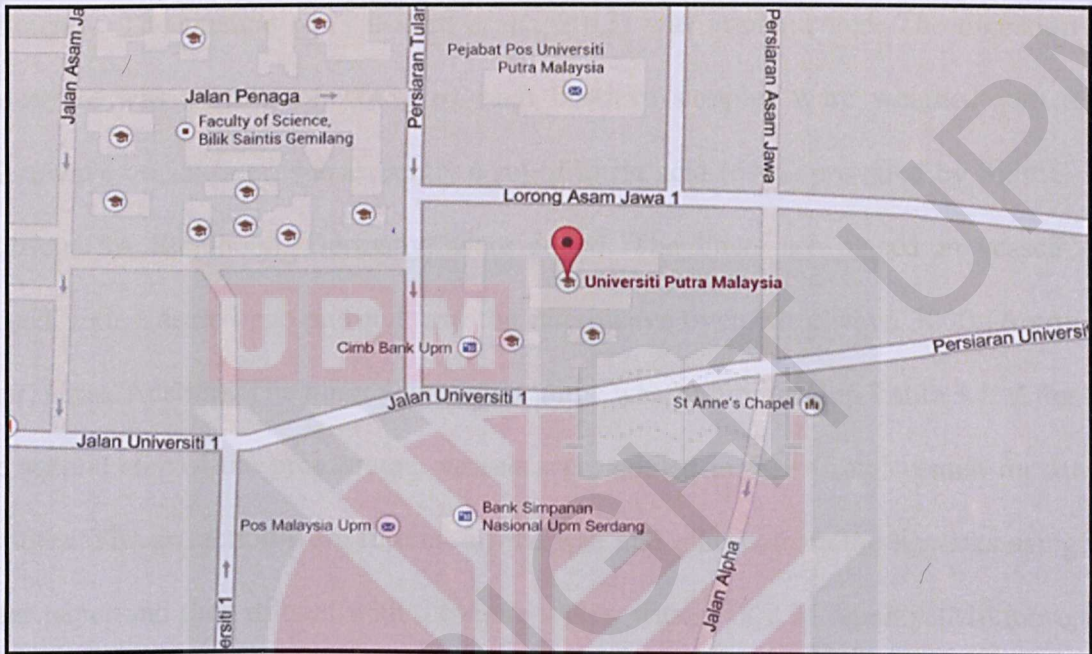
**p** = estimated percentage of women applying lipsticks (58%) (DASH, 2006)

**m** = margin of error at 5% (standard value of 0.05)

$$N = \frac{1.96^2 \times 0.58(1-0.58)}{0.05^2}$$

$$= 374 \text{ respondents}$$

= 374 respondents



**Figure 3.1. Universiti Putra Malaysia (UPM)**

Three hundred questionnaires were distributed to the female staffs that applied lipsticks around UPM. Due to the time constraint and lack of resource to reach these female staffs of UPM the response rate of the respondents in answering the questionnaire was only around 72% which was 215 respondents over 300 respondents supposedly.

### 3.2.2 Sample Preparation through Microwave Digestion Technique

The US FDA in-house validated method of Microwave digestion technique is chosen for the heavy metals determination technique for its high lead recovery and efficiency. The method of (Piccinini *et al.*, 2013) was implemented. The digestion procedure was as follows: 0.4 g of each lipsticks samples were weighed into a microwave vessel liner, subsequently 6 ml of nitric acid (69%) provided by Sigma-Aldrich (SA Steinheim, Germany) were added. The liners are placed in vessels, closed with a sealed cap and put into the microwave oven Multiwave 3000 (Anton Paar, Graz, Austria). The microwave programme was summarised in Table 3.1. After the second step of the programme, vessels were cooled to 50 ° C and vented for 10 minutes. The extraction were filtered for the wax and glitters from the lipsticks using filter paper and then diluted with ultrapure water, water, 18.2 M. quality (Millipore, Vimodrone (MI), Italy) to the final volume of 50 ml (Qureshi, 2011). There might be product loss in filtration of the glitters and waxes since metals could still be bound to waxes. To overcome this limitation, each sample was being analyzed triplicate, together with two method blanks. Acid washing was done for cleaning the vessel of microwave oven before being used again for the next sample batch.

Table 3.1 Microwave Programme Used For Samples' Digestion

Step	Ramp (min)	Temperature ( ° C)	Hold (min)	Cold (min)
1	15	130	20	
2	15	200	20	
3	-	50	-	10

### 3.2.3 Sample Analysis using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES)

The thermo Scientific iCAP 6000 Series ICP\_OES was used to analyse the lipsticks sample (N=15). iCAP 6000 Series used the intuitive and powerful iTEVA Software to run the analysis. The calibration point for the ICP-OES is 0.005, 0.05 ppm, 0.1 ppm, 0.5 ppm, 1 ppm, 3 ppm and 5 ppm (Madison, 2005). The 0.25 ppm standard solution is used as a quality control. Nitric acid of 69% was prepared through the microwave digestion technique serving as a calibration blank. The calibration blank were firstly analysed before the samples. The weight of the lipsticks sample (0.4 g) and its final volume of extraction solution (50 mL) information had been included in the system of ICP-OES.

### 3.2.3 Quality Assurance (QA) and Quality Control (QC)

For the lipsticks samples processed, a method blank was carried throughout the entire sample preparation and analytical process (US EPA, 1996). These blanks will be useful in determining if samples are being contaminated. A recovery samples for the digestion technique were prepared. The organic lip balm was divided into two group: the first one ( $A_1$ ) were spike with 0.25 ppm standard before the digestion while the second group ( $A_2$ ) was spike with 0.25 ppm standard after the digestion. The method extraction efficiency was high as confirmed by the recovery values obtained for spike organic lip balm materials that were analyzed. The confidence intervals for the means, calculated at 95% probability, were equal to 91% for chromium (Cr), 94% for cadmium (Cd) and 77% for lead (Pb) respectively. The formula of gaining these recovery values was as shown below:

$$\text{Recovery (\%)} = \frac{A_1 - \text{blank concentration}}{A_2 - \text{blank concentration}} \times 100\% \quad (\text{Eq 3.2})$$

The sediment that was present in any of the digested samples was filtered before the extraction was diluted into 50 ml final concentration with ultrapure water (US EPA1996). All sample containers were prewashed with detergents, acids, and water. Acid wash was done on the vessel liner to prevent any contamination from the previous lipstick samples digested. The vessel liners were left overnight in 10% acid water to corrode any sediment left or sticking on the vessel liners wall (Madison, 2005).

Identical samples are analyzed three times within one-day's acquisition sequence. A lipstick sample was triplicate in order to gain a more precise estimation of the data (US EPA, 1996).

### 3.2.4 Quantitative Health Risk Assessments

Hazard Quotient (HQ) is the ratio of the average daily dose (ADD) to the reference dose (RfD). It is a measure of the possibility of seeing chronic, non-carcinogenic health effects among the exposed respondents were calculated. The formula is given below:

$$\text{For Ingestion: } HQ = \frac{ADD}{RfD}, \quad \text{Eq.(3.3)}$$

Table 3.2 Interpretation of Hazard Quotient

Name	Symbol	Unit	Value	Reference
Hazard Quotient	HQ	-	$\frac{ADD}{RfD}$	This study
Average Daily Dose	ADD	mg/kg-day	$\frac{C_f \times Ri \times Fi \times EF \times ED}{BW \times AT}$	This study
Reference Dose	RfD	mg/kg-day	refer Table 3.3	refer Table 3.3

Table 3.3 Information of the limit of Reference Dose

Heavy metal	UF	MF	RfD	Unit	References
Lead	-	-	10	mg/kg/day	Health Canada, 2011
Cadmium	10	1	3	mg/kg/day	Intergrated Risk Information System, 2013
Chromium	300	3	3	mg/kg/day	Intergrated Risk Information System, 2013

ADD can be calculated for ingestion of heavy metals exposure by using this formula:

$$\begin{aligned}
 \text{ADD} &= \frac{C_f \times R_i \times F_i \times E_f \times E_d}{BW \times AT} && \text{Eq.(3.4)} \\
 &= \frac{C_f \times 1.23 \times 0.010 \times 2.35 \times 6.71}{57.85 \times 7300}
 \end{aligned}$$

Table 3.4 Interpretation of Average Daily Dose (ADD)

Name	Acro- nyms	Unit	Value	References
Average daily dose of pollutant through ingestion of food	<b>ADD</b>	mg/kg.day	$\frac{C_f \times R_i \times F_i \times E_f \times E_d}{BW \times AT}$ $\frac{C_f \times 1.23 \times 0.010 \times 2.35 \times 6.71}{57.85 \times 7300}$	This study
Pollutant concentration in the lipsticks sample	<b>C<sub>F</sub></b>	mg/kg	The value of heavy metals in the lipsticks sample	This study
Food item ingestion rate	<b>R<sub>i</sub></b>	g/day	Cosmetic Co. (1.23)	EPA, 2013
Fraction ingested from contaminated source	<b>F<sub>i</sub></b>	g/day	0.010	EPA, 2013
Exposure frequency	<b>E<sub>F</sub></b>	No. of applicatio n/day	2.35	EPA, 2013
Exposure duration	<b>E<sub>D</sub></b>	Year	The mean duration of the women respondents starts applied lipsticks until	This study

			present day	
			(6.71 years)	
			Mean of the respondents'	
Body weight	<b>BW</b>	Kg	body weight that taking part in the survey	This study
			(57.85)	
Averaging time	<b>AT</b>	Day	20 years/7300	EPA, 2013

HQ (Hazard Quotient) compares the magnitude of ADD (Estimated Average Daily Dose) with that of RfD (Reference Dose). If HQ is larger than 1, i.e., if ADD exceeds RfD, there is risk. If HQ is smaller than 1, i.e., if ADD does not exceed RfD (NITE, 2011).

For the carcinogenic health effects of the Cadmium and Chromium exposure form the usage of lipsticks, the LCR is the formula to measure the possibility of developing this health effects. The formula is given below:

$$\text{For Ingestion: } \text{LCR} = \text{LADD} \times \text{CSF}, \quad \text{Eq.(3.5)}$$

Table 3.5 Interpretation of Lifetime Excess Cancer Risk (LCR);

Name	Acronyms	Unit	Value	Reference
Lifetime Excess Cancer Risk	LCR	-	<b>LADD x CSF</b>	This study
Lifetime Average Daily Dose	LADD	mg/kg-day	$\frac{C_f \times R_i \times F_i \times E_f \times E_d}{BW \times AT}$	This study
Cancer Slope Factor	CSF	mg/kg-day	refer Table 3.6	refer Table 3.6

Table 3.6 Information of the Cancer Slope Factor (CSF)

Heavy metal	CSF	Unit	References
Cadmium	6.1	mg/kg/day	Intergrated Risk Information System, 2013
Chromium	42	mg/kg/day	Intergrated Risk Information System, 2013

The LADD can be calculated for ingestion of heavy metals exposure by using this formula:

$$LADD = \frac{C_f \times R_i \times F_i \times E_f \times E_d}{BW \times AT}, \quad \text{Eq.(3.6)}$$

$$= \frac{C_f \times 1.23 \times 0.010 \times 2.35 \times 6.71}{57.85 \times 25550}$$

Table 3.7 Interpretation of Average Daily Dose (LADD),

Name	Acronyms	Unit	Value	References
Lifetime average daily dose of pollutant through ingestion of food	<b>LAD</b> <b>D</b>	mg/kg.day	$\frac{C_f \times R_i \times F_i \times E_f \times E_d}{B_w \times A_t}$ $\frac{C_f \times 1.23 \times 0.010 \times 2.35 \times 6.71}{57.85 \times 25550}$	This study
Pollutant concentration in the lipsticks sample	<b>C<sub>F</sub></b>	mg/kg	The value of heavy metals in the lipsticks sample	This study
Food item ingestion rate	<b>R<sub>i</sub></b>	g/day	Cosmetic Co. -1.23	EPA, 2013
Fraction ingested from contaminated source	<b>F<sub>i</sub></b>	g/day	0.010	EPA, 2013
Exposure frequency	<b>E<sub>F</sub></b>	No. of application	2.35	EPA, 2013

---

			/ day		
Exposure duration	<b>ED</b>	Year	The mean duration of the women respondents starts applied lipsticks until present day (6.71 years)		This study
Body weight	<b>BW</b>	Kg	Mean of the respondents' body weight that taking part in the survey (57.85)		This study
Averaging time	<b>AT</b>	Day	70 years / 25550days		EPA, 2013

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### 3.3 Statistical Analysis

**Descriptive statistical parameters** such as mean and SD are use to describe the heavy metal concentrations in 15 lipstick samples ( Al-Saleh & Al-Enazi, 2011).

**One Way Analysis of Variance (ANOVA)** to determine the differences of the heavy metals mean concentration among low, intermediate, and high price of lipsticks at a significance level of  $p < 0.05$  ( Al-Saleh & Al-Enazi, 2011).

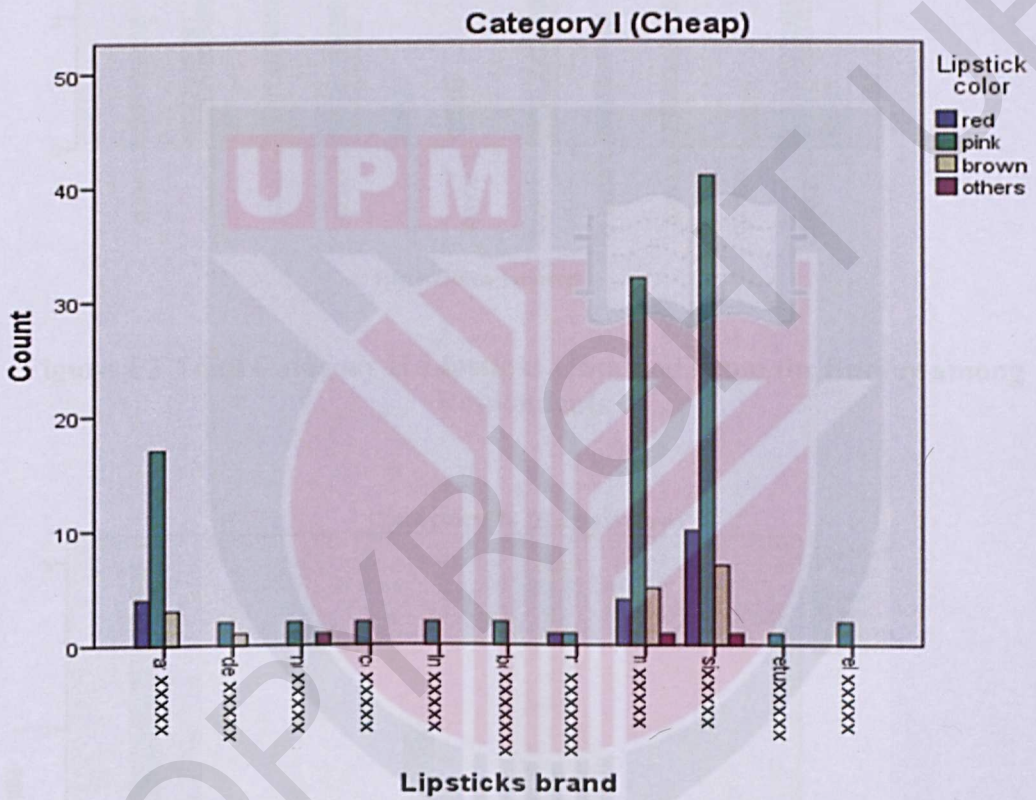
**Kruskal-Wallis One Way Analysis of Variance (ANOVA)** to determine the differences of the heavy metals mean concentration among low, intermediate, and high price of lipsticks at a significance level of  $p < 0.05$  (I. Al-Saleh & Al-Enazi, 2011)

#### 4. RESULT



## CHAPTER 4

### 4. RESULT



**Figure 4.1 Total Category I Lipsticks Obtained From the Survey among Respondents**

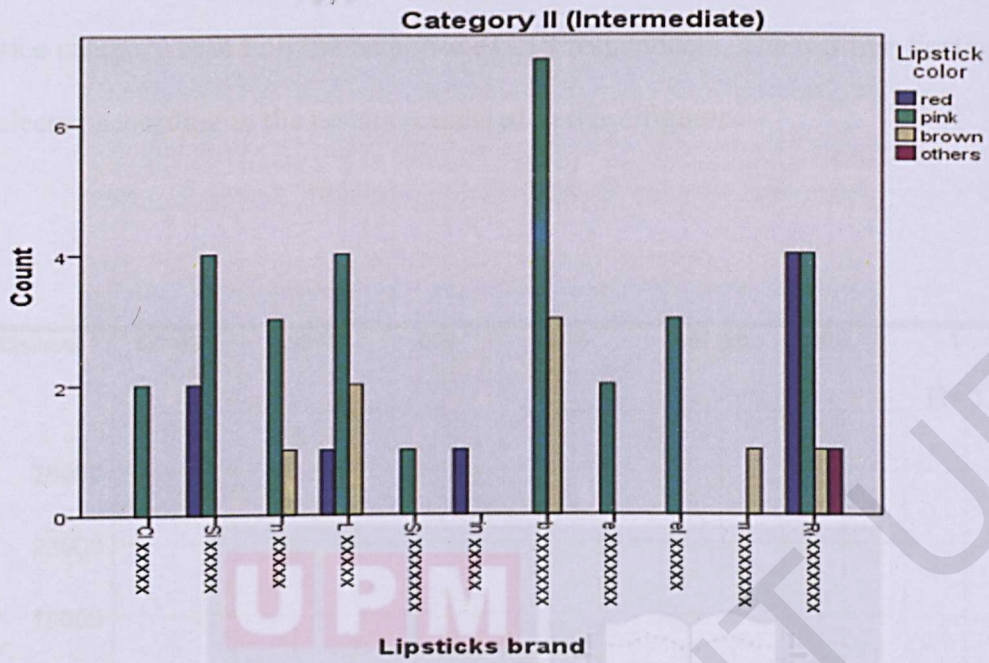


Figure 4.2 Total Category II Lipsticks Obtained From the Survey among Respondents

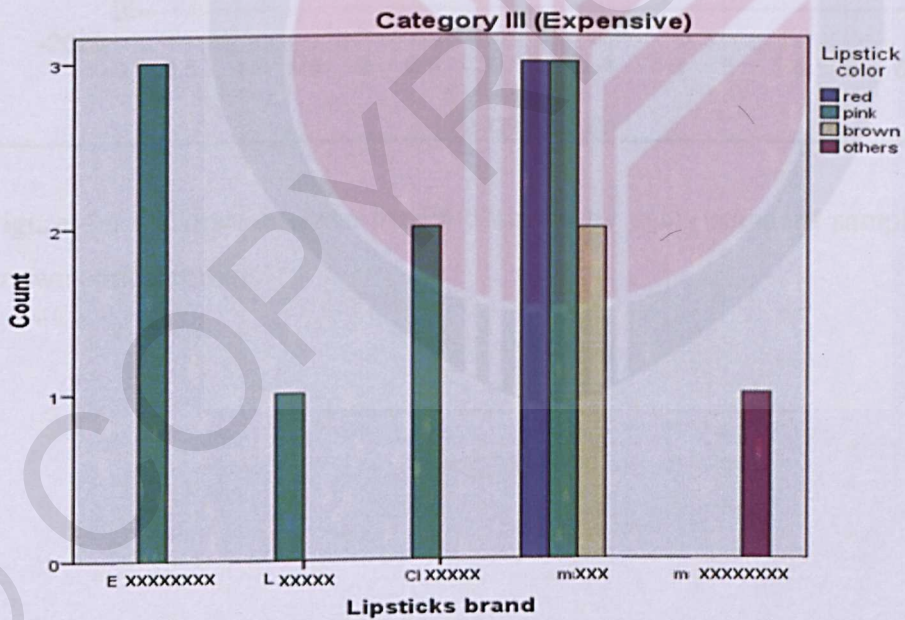


Figure 4.3 Total Category III Lipsticks Obtained From the Survey among Respondents

Figure 4.1 to 4.3 showed that the selection of the lipstick samples in each price category based on the response of 215 respondents. The top five lipsticks were selected according to the results presented in these figures.

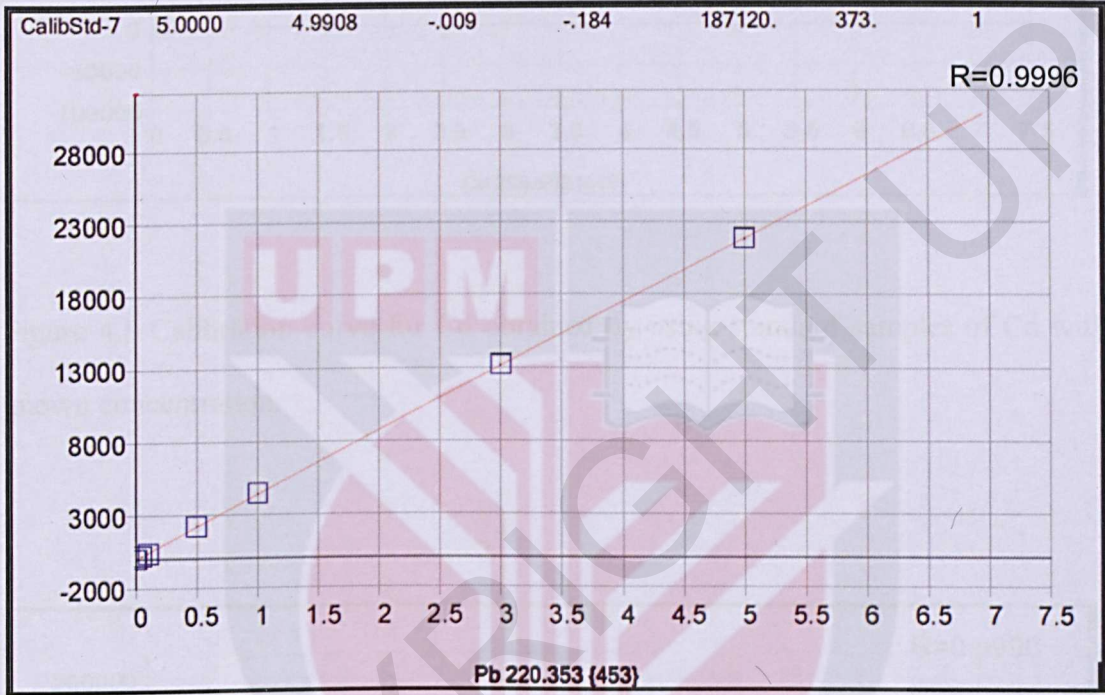


Figure 4.4 Calibration curve for Pb obtained by using standard samples of Pb with known concentration.



Figure 4.5 Calibration curve for Cd obtained by using standard samples of Cd with known concentration.

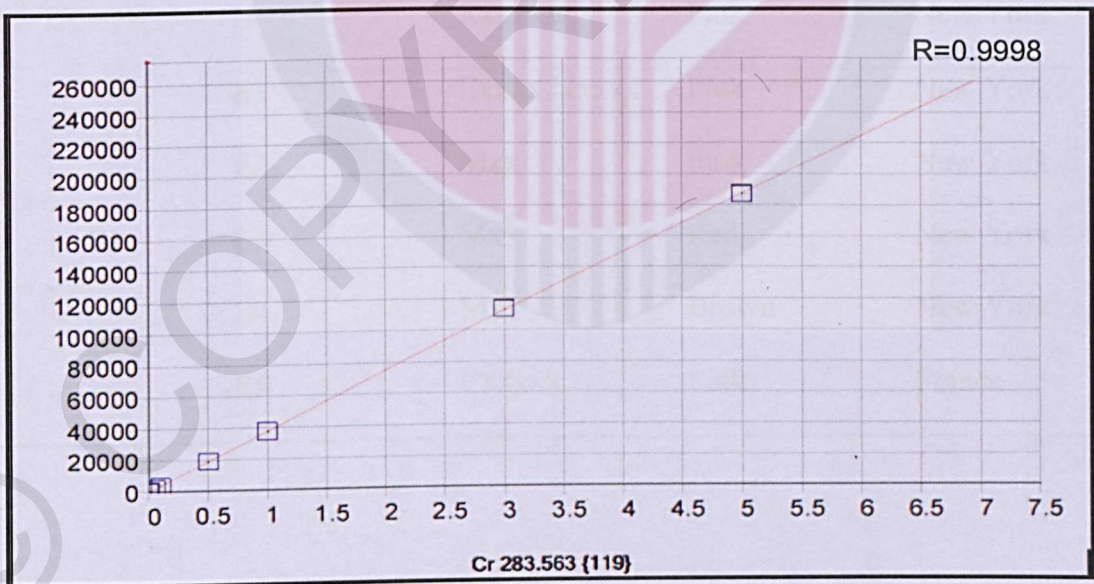


Figure 4.6. Calibration curve for Cr obtained by using standard samples of Cr with known concentration.

Table 4.1. The country of production of the lipsticks samples

Price Category	Lipsticks code	Brand	Colour	Country of Production
I	C1	Sxxxx Gxxx	Red	Malaysia
	C2	Sxxxx Gxxx	Pink	Malaysia
	C3	Sxxxx Gxxx	Nude	Malaysia
	C4	Mxxxxxxxx	Pink	New York
	C5	Axxx	Pink	Malaysia
II	I1	Sxxxxx sxxx	Pink	Korea
	I2	Lxxxxx pxxxx	Pink	France
	I3	Bxxx sxxx	Pink	United Kingdom
	I4	Rxxxxx	Red	New York
	I5	Rxxxxx	Pink	New York
III	E1	Exxxx lxxxxx	Pink	New York
	E2	Mxx	Pink	New York
	E3	Mxx	Red	New York
	E4	Mxx	Brown	New York
	E5	Cxxxxx	Pink	France

Table 4.2 Heavy metals concentrations in the top popular category I lipsticks used by UPM female staffs.

Category	Price	Code	Brand	Color	Mean±SD (mg/kg)		
					Pb	Cd	Cr
I					(N=3)		
		C1	Sxxxx Gxxx	Red	2.52 ± 0.041	0.334 ± 0.053	0.508 ± 0.044
		C2	Sxxxx Gxxx	Pink	3.742 ± 0.348	0.268 ± 0.0001	0.578 ± 0.187
		C3	Sxxxx Gxxx	Nude	4.421 ± 5.769	0.028 ± 0.029	2.061 ± 1.168
		C4	Mxxxxxxxxx	Pink	4.028 ± 5.362	0.014 ± 0.008	1.434 ± 0.650
		C5	Axxx	Pink	1.725 ± 1.497	0.084 ± 0.054	0.623 ± 0.353

The highest mean concentration of Pb in the price category I was 4.421 ± 5.769 mg/kg in brown Sxxxx Gxxx lipstick. The lowest mean concentration of Pb was 1.725 ± 1.497 mg/kg in the pink colour lipsticks of Axxx brand. The red Sxxxx Gxxx lipstick had highest concentration of Cd in this category which was 0.334 ± 0.053 mg/kg, while the brown Sxxxx Gxxx lipsticks had the minimum mean concentration of Cd which is 0.028 ± 0.029 mg/kg. The highest Cr mean

concentration was  $2.061 \pm 1.168$  mg/kg in brown Sxxxx Gxxx lipstick while the lowest mean concentration was  $0.508 \pm 0.044$  mg/kg in red Sxxxx Gxxx lipsticks.

Table 4.3 Heavy metals concentrations in the top popular category II lipsticks used by UPM female staffs.

Category	Price	Code	Brand	Color	Mean±SD (mg/kg)		
					(n=3)		
					Pb	Cd	Cr
I1			Sxxxxx	Pink	$1.956 \pm$	$0.094 \pm$	$0.796 \pm$
I2			Lxxxxx	Pink	1.637	0.066	0.451
I3	II	I3	Bxxx sxxx	Pink	$0.901 \pm$	$0.186 \pm$	$0.479 \pm$
I4			Rxxxxx	Red	1.046	0.087	0.273
I5			Rxxxxx	Pink	$1.101 \pm$	$0.074 \pm$	$2.024 \pm$
					0.954	0.044	1.144
					$0.771 \pm$	$0.038 \pm$	$0.242 \pm$
					0.635	0.022	0.149
					$1.113 \pm$	$0.033 \pm$	$0.241 \pm$
					0.989	0.028	0.138

The highest mean concentration of Pb in the price category II was  $1.956 \pm 1.637$  mg/kg in pink Sxxxxx Sxxx lipstick. The lowest mean concentration of Pb was

0.771 ± 0.635 mg/kg in the red colour lipsticks of Rxxxxx brand. The pink Lxxxxx Pxxxx lipstick had highest concentration of Cd in this category which was 0.186 ± 0.087 mg/kg, while the Pink Rxxxxx lipsticks had the lowest mean concentration of Cd which was 0.033 ± 0.028 mg/kg. The highest mean concentration of Cr was 2.024 ± 1.144 mg/kg in pink Lxxxxx Pxxxx lipstick while the lowest mean concentration was 0.241 ± 0.138 mg/kg in pink Rxxxxx lipsticks.

Table 4.4 Heavy metals concentrations in the top popular category III lipsticks used by UPM female staffs.

Category	Price	Code	Brand	Color	Mean ± SD (mg/kg)		
					(n=3)		
					Pb	Cd	Cr
III		E1	Exxxxx	Pink	1.104 ± 0.958	0.070 ±	1.211 ±
			Lxxxxx			0.059	0.682
		E2	Mxx	Pink	3.370 ± 4.681	0.057 ±	2.503 ±
						0.050	1.437
		E3	Mxx	Red	3.753 ± 5.753	0.005 ±	0.904 ±
						0.008	1.470
		E4	Mxx	Brown	15.440 ± 22.079	0.092 ±	0.683 ±
						0.053	0.471
		E5	Cxxxxx	Pink	2.175 ± 0.418	0.0667 ±	0.848 ±
						0.006	0.076

The highest mean concentration of Pb in the price category III was  $15.440 \pm 22.079$  mg/kg in brown Mxx lipstick. The lowest mean concentration of Pb was  $1.104 \pm 0.958$  mg/kg in the pink colour lipsticks of Exxxx Lxxxxx brand. The brown Mxx lipstick had highest concentration of Cd in this category which was  $0.092 \pm 0.053$  mg/kg, while the red Mxx lipsticks had the lowest mean concentration of Cd which was  $0.005 \pm 0.008$  mg/kg. The highest mean concentration of Cr was  $2.503 \pm 1.437$  mg/kg in pink Mxx lipstick while the lowest mean concentration was  $0.683 \pm 0.471$  mg/kg in red Mxx lipsticks.

Table 4.5 Comparison of Heavy Metals Concentration in Lipsticks of Different Price Categories (n=15).

Price Category	Total samples	Lipsticks		
		Mean $\pm$ SD (mg/kg)		
		Pb	Cd	Cr
I	5	$3.287 \pm 1.126$	$0.146 \pm 0.146$	$1.041 \pm 0.683$
II	5	$1.168 \pm 0.463$	$0.085 \pm 0.062$	$0.756 \pm 0.744$
III	5	$5.168 \pm 5.836$	$0.058 \pm 0.032$	$1.230 \pm 0.737$

The average concentration of Pb mean concentration in the category I lipsticks samples (n=5) was  $3.287 \pm 1.126$  mg/kg. The average concentration of Cd in this price category (n=5) was  $0.085 \pm 0.062$  mg/kg while the Cr average concentration in these lipsticks samples (n=5) was  $1.041 \pm 0.683$  mg/kg.

The average concentration of Pb mean concentration in the category II lipsticks samples (n=5) was  $1.168 \pm 0.463$  mg/kg. The average concentration of Cd in this price category (n=5) was  $0.146 \pm 0.146$  mg/kg the Cr average concentration in these lipsticks samples (n=5) was  $0.756 \pm 0.744$  mg/kg.

The average concentration of Pb mean concentration in the category III lipsticks samples (n=5) was  $5.168 \pm 5.836$  mg/kg. The average concentration of Cd in this price category (n=5) was  $0.058 \pm 0.032$  mg/kg while the Cr average concentration in these lipsticks samples (n=5) was  $1.230 \pm 0.737$  mg/kg.

Table 4.6 The test of normality the occurrence of Lead (Pb) between the price categories of the lipsticks samples (n=15).

	Price Category	Shapiro-Wilk
		P
Mean concentration	I	0.508
	II	0.118
	III	0.016

Based on the table above, p values of each category were smaller than 0.05. It indicates that the data was not normally distributed. Therefore, Independent Samples Kruskal-Wallis Test was used.

Table 4.7 The Independent Samples Kruskal-Wallis test on differences of the occurrence of Lead (Pb) between the price categories of the lipsticks samples (n=15).

Price Category	N	Med	IQR	Mean Rank	H	P
I	5	3.742	2.102	10.60		
II	5	1.101	0.698	3.60	7.340	0.025
III	5	3.370	7.957	9.80		

The following null and alternative hypotheses were used;  $H_0$ : The Pb mean concentration in lipsticks is not significantly different between the I, II, and III price category of the lipsticks,  $H_1$ : The Pb mean concentration in lipsticks is significantly different between the I, II, and III price category of the lipsticks. The statistical data showed the value of H was 7.340 and p value was 0.025. The p value is smaller than 0.05, thus the null hypothesis is rejected. Therefore, there is a significant difference of Pb mean concentration level between the I, II, and III price category of the lipsticks. The mean rank in the table above showed that the price category I had the highest mean concentration of Pb which was 10.60 mg/kg compared to 9.80 mg/kg in category III followed by 3.60 mg/kg in category II. The price category I recorded the highest median score which was 3.742 mg/kg compared to 3.370 mg/kg in category III and 1.101 mg/kg in category II. The Mann-Whitney U Test was done to determine the significant difference between two different price categories at  $p < 0.017$ .

Table 4.8 The Mann-Whitney U test on differences of the occurrence of Lead (Pb) between the price categories I and II (n=10).

	Lipsticks Categories	N	Mean Rank	Man-	Z	P
				Whitney U		
Pb Concentration	I	5	7.80			
	II	5	3.20	1.000	2.402	0.016
	Total	10				

The following null and alternative hypotheses were used;  $H_0$ : The Pb mean concentration in lipsticks is not significantly different between the I and II price category of the lipsticks,  $H_1$ : The Pb mean concentration in lipsticks is significantly different between the I and II price category of the lipsticks. The statistical data showed the value of Z was 2.402 and p value was 0.016. The p value is smaller than 0.017, thus the null hypothesis is rejected. Therefore, there is a significant difference of Pb mean concentration level between the I and II price category of the lipsticks. The mean rank in the table above showed that the price category I had the highest mean concentration of Pb which was 7.8 mg/kg compared to 3.2 mg/kg in category II.

Table 4.9 The Mann-Whitney U test on differences of the occurrence of Lead (Pb) between the price categories II and III (n=10).

	Lipsticks Categories	N	Mean Rank	Man-Whitney U		
				Z	P	U
Pb Concentration	II	5	3.40			
	III	5	7.60	2.000	2.193	0.028
	Total	10				

The following null and alternative hypotheses were used;  $H_0$ : The Pb mean concentration in lipsticks is not significantly different between the II and III price category of the lipsticks,  $H_1$ : The Pb mean concentration in lipsticks is significantly different between II and III price category of the lipsticks. The statistical data showed the value of Z was 2.193 and p value was 0.028. The p value is smaller than 0.017, thus the null hypothesis is rejected. Therefore, there is a significant difference of Pb mean concentration level between the I and II price category of the lipsticks. The mean rank in the table above showed that the price category III had the highest mean concentration of Pb which was 7.6 mg/kg compared to 3.4 mg/kg in category II.

Table 4.10 The Mann-Whitney U test on differences of the occurrence of Lead (Pb) between the price categories I and III (n=10).

	Lipsticks Categories	N	Mean Rank	Man-Whitney U		
				U	Z	P
Pb Concentration	I	5	5.80			
	III	5	5.20	11.000	0.313	0.754
	Total	10				

The following null and alternative hypotheses were used;  $H_0$ : The Pb mean concentration in lipsticks is not significantly different between the I and III price category of the lipsticks,  $H_1$ : The Pb mean concentration in lipsticks is significantly different between I and III price category of the lipsticks. The statistical data showed the value of Z was 0.313 and p-value was 0.754. The p value is bigger than 0.017, thus the null hypothesis is rejected. Therefore, there is a significant different of Pb mean concentration level between the I and III price category of the lipsticks. The mean rank in the table above showed that the price category I had close mean concentration of Pb which was 5.8 mg/kg to 5.2 mg/kg in category III.

Table 4.11 The test of normality the occurrence of Cadmium (Cd) between the price categories of the lipsticks samples (n=15).

	Price Category	Shapiro-Wilk
		P
Mean concentration	I	0.219
	II	0.265
	III	0.330

Based on the table above, p values of each category were more than 0.05. It indicates that the data was normally distributed. Therefore, One Way ANOVA test was used.

Table 4.12 The One Way Analysis of Variance (ANOVA) test on differences of the occurrence of Cadmium (Cd) between the price categories of the lipsticks samples (n=15).

Price Category	N	Mean	SD	F	P
I	5	0.146	0.146	1.150	0.349
II	5	0.009	0.062		
III	5	0.058	0.032		

The Levene Statistics showed the value of F was 11.139, while the p value was 0.002. p value was smaller than 0.05, thus the assumption on homogeneity has not been violated. The following null and alternative hypothesis were used;  $H_0$ : The Cd mean concentration in lipsticks is not significantly different between the I, II, and III price category of the lipsticks,  $H_1$ : The Cd mean concentration in lipsticks is significantly different between the I, II, and III price category of the lipsticks. For the ANOVA test, the statistical data showed the value of F was 1.150 and p value was 0.349. The p value is bigger than 0.05, thus the null hypothesis is failed to be rejected. Therefore, there are no significant different of Cd mean concentration level between the I, II, and III price category of the lipsticks.

Table 4.13 The test of normality the occurrence of Chromium (Cr) between the price categories of the lipsticks samples (n=15).

	Price Category	Shapiro-Wilk
		P
Mean concentration	I	0.121
	II	0.061
	III	0.049

Based on the table above, p values of each category were smaller than 0.05. It indicates that the data was not normally distributed. Therefore, the Independent Samples Kruskal-Wallis test was used.

Table 4.14 The Independent Samples Kruskal-Wallis test on differences of the occurrence of Chromium (Cr) between the price categories of the lipsticks samples (n=15).

Price Category	N	Med	IQR	Mean Rank	H	P
I	5	0.623	1.2045	8.20		
II	5	0.479	1.1684	5.40	3.140	0.208
III	5	0.904	1.0913	10.40		

The following null and alternative hypotheses were used;  $H_0$ : The Cr mean concentration in lipsticks is not significantly different between the I, II, and III price category of the lipsticks,  $H_1$ : The Cr mean concentration in lipsticks is significantly different between the I, II, and III price category of the lipsticks. The statistical data showed the value of H was 3.140 and p value was 0.208. The p value is bigger than 0.05, thus the null hypothesis is fail to be rejected. Therefore, there is no significant different of Cr mean concentration level between the I, II, and III price category of the lipsticks. The mean rank in the table above showed that the price category III had the highest mean concentration of Cr which was 10.40 mg/kg compared to 8.20 mg/kg in category I followed by 5.40 mg/kg in category II. The price category I recorded the highest median score which was 0.904 mg/kg compared to 0.623 mg/kg in category II and 0.479 mg/kg in category III.

Table 4.15 Non-carcinogenic health risk assessments for the exposure towards Lead (Pb) concentration in the lipsticks sample based on the lipsticks brand (n=15).

Lipsticks code	Brand	Pb Mean		
		concentration (mg/kg)	ADD	HQ
C1	Sxxxx Gxxx	2.520	$1.16 \times 10^{-6}$	$1.16 \times 10^{-7}$
C2	Sxxxx Gxxx	3.742	$1.72 \times 10^{-6}$	$1.72 \times 10^{-7}$
C3	Sxxxx Gxxx	4.421	$2.03 \times 10^{-6}$	$2.03 \times 10^{-7}$
C4	Mxxxxxxxxx	4.028	$1.85 \times 10^{-6}$	$1.85 \times 10^{-7}$
C5	Axxx	1.725	$7.9 \times 10^{-7}$	$7.9 \times 10^{-8}$
I1	Sxxxxx sxxx	1.956	$9.0 \times 10^{-7}$	$9.0 \times 10^{-8}$
I2	Lxxxxx pxxxx	0.901	$4.1 \times 10^{-7}$	$4.1 \times 10^{-8}$
I3	Bxxx sxxx	1.101	$5.1 \times 10^{-7}$	$5.1 \times 10^{-8}$
I4	Rxxxxx	0.771	$3.5 \times 10^{-7}$	$3.5 \times 10^{-8}$
I5	Rxxxxx	1.113	$5.1 \times 10^{-7}$	$5.1 \times 10^{-8}$
E1	Exxxx lxxxx	1.104	$5.1 \times 10^{-7}$	$5.1 \times 10^{-8}$
E2	Mxx	3.370	$1.55 \times 10^{-6}$	$1.55 \times 10^{-7}$
E3	Mxx	3.753	$1.72 \times 10^{-6}$	$1.72 \times 10^{-7}$
E4	Mxx	15.440	$7.09 \times 10^{-6}$	$7.09 \times 10^{-7}$
E5	Cxxxxx	2.175	$1.00 \times 10^{-6}$	$1.00 \times 10^{-7}$



Table 4.16 Non-carcinogenic health risk assessments for the exposure towards Cadmium (Cd) concentration in the lipsticks sample based on the lipsticks brand (n=15).

Lipsticks code	Brand	Cd Mean		
		concentration (mg/kg)	ADD	HQ
C1	Sxxxx Gxxx	0.3339	$1.5 \times 10^{-7}$	$5.1 \times 10^{-8}$
C2	Sxxxx Gxxx	0.2676	$1.2 \times 10^{-7}$	$4.1 \times 10^{-8}$
C3	Sxxxx Gxxx	0.0283	$1.3 \times 10^{-8}$	$4.0 \times 10^{-9}$
C4	Mxxxxxxxxx	0.0143	$6.6 \times 10^{-9}$	$2.0 \times 10^{-9}$
C5	Axxx	0.0840	$4.0 \times 10^{-7}$	$1.3 \times 10^{-8}$
I1	Sxxxxx sxxx	0.0944	$4.3 \times 10^{-8}$	$1.4 \times 10^{-8}$
I2	Lxxxxx pxxxx	0.1862	$8.5 \times 10^{-8}$	$2.9 \times 10^{-8}$
I3	Bxxx sxxx	0.0742	$3.4 \times 10^{-8}$	$1.1 \times 10^{-8}$
I4	Rxxxxx	0.0380	$1.7 \times 10^{-8}$	$6.0 \times 10^{-9}$
I5	Rxxxxx	0.0325	$1.5 \times 10^{-8}$	$5.0 \times 10^{-9}$
E1	Exxxxx lxxxxx	0.0695	$3.1 \times 10^{-8}$	$1.1 \times 10^{-8}$
E2	Mxx	0.0574	$2.6 \times 10^{-8}$	$9.0 \times 10^{-9}$
E3	Mxx	0.0049	$2.3 \times 10^{-9}$	$7.5 \times 10^{-10}$
E4	Mxx	0.0921	$4.2 \times 10^{-8}$	$1.4 \times 10^{-8}$
E5	Cxxxxx	0.0667	$3.1 \times 10^{-8}$	$1.0 \times 10^{-8}$

Generally, The Hazard Quotient (HQ) value for Cd exposure towards the lipsticks consumer showed HQ less than 1 as the results on all lipsticks brand. It indicates a sign of clearly acceptable non-carcinogenic health risk.



Table 4.17 Non-carcinogenic health risk assessments for the exposure towards Chromium (Cr) concentration in the lipsticks sample based on the lipsticks brand (n=15).

Lipsticks code	Brand	Cr Mean		
		concentration (mg/kg)	ADD	HQ
C1	Sxxxx Gxxx	0.5077	$2.3 \times 10^{-7}$	$7.8 \times 10^{-8}$
C2	Sxxxx Gxxx	0.5778	$2.7 \times 10^{-7}$	$8.8 \times 10^{-8}$
C3	Sxxxx Gxxx	2.0610	$9.5 \times 10^{-7}$	$3.2 \times 10^{-7}$
C4	Mxxxxxxxx	1.4335	$6.6 \times 10^{-7}$	$2.2 \times 10^{-7}$
C5	Axxx	0.6234	$2.9 \times 10^{-7}$	$9.5 \times 10^{-8}$
I1	Sxxxxx sxxx	0.7956	$3.7 \times 10^{-7}$	$1.2 \times 10^{-7}$
I2	Lxxxxx pxxxx	0.4785	$2.2 \times 10^{-7}$	$7.3 \times 10^{-8}$
I3	Bxxx sxxx	2.0240	$9.3 \times 10^{-7}$	$3.1 \times 10^{-7}$
I4	Rxxxxx	0.2424	$1.1 \times 10^{-7}$	$3.7 \times 10^{-8}$
I5	Rxxxxx	0.2405	$1.1 \times 10^{-7}$	$3.7 \times 10^{-8}$
E1	Exxxx lxxxxx	1.2105	$5.6 \times 10^{-7}$	$1.9 \times 10^{-7}$
E2	Mxx	2.5030	$1.2 \times 10^{-7}$	$3.8 \times 10^{-7}$
E3	Mxx	0.9042	$4.2 \times 10^{-7}$	$1.4 \times 10^{-7}$
E4	Mxx	0.6832	$3.1 \times 10^{-7}$	$1.1 \times 10^{-7}$
E5	Cxxxxx	0.8476	$3.9 \times 10^{-7}$	$1.3 \times 10^{-7}$

Generally, The Hazard Quotient (HQ) value for Pb exposure towards the lipsticks consumer showed HQ less than 1 as the results on all lipsticks brand. It indicates a sign of clearly acceptable non-carcinogenic health risk.

Lipstick code	Brand	Concentration (mg/kg)	LAAD	LCR
C1	Shera-Gala	0.407	$4.7 \times 10^{-5}$	$2.7 \times 10^{-5}$
C2	Shera-Gala	0.279	$3.3 \times 10^{-5}$	$1.9 \times 10^{-5}$
C3	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C4	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C5	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C6	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C7	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C8	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C9	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C10	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C11	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C12	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C13	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C14	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C15	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C16	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C17	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C18	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C19	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$
C20	Shera-Gala	0.410	$4.9 \times 10^{-5}$	$2.8 \times 10^{-5}$

Table 4.18 Carcinogenic health risk assessments for the exposure towards Cadmium (Cd) concentration in the lipsticks sample based on the lipsticks brand (n=15).

Lipsticks code	Brand	Cd Mean		
		concentration (mg/kg)	LADD	LCR
C1	Sxxxx Gxxx	0.5077	$4.4 \times 10^{-8}$	$2.7 \times 10^{-7}$
C2	Sxxxx Gxxx	0.5778	$3.5 \times 10^{-8}$	$2.1 \times 10^{-7}$
C3	Sxxxx Gxxx	2.0610	$3.7 \times 10^{-9}$	$2.3 \times 10^{-8}$
C4	Mxxxxxxxx	1.4335	$1.9 \times 10^{-9}$	$1.1 \times 10^{-8}$
C5	Axxx	0.6234	$1.1 \times 10^{-8}$	$6.7 \times 10^{-8}$
I1	Sxxxxx sxxx	0.7956	$1.2 \times 10^{-8}$	$7.6 \times 10^{-8}$
I2	Lxxxxx pxxxx	0.4785	$2.4 \times 10^{-8}$	$1.5 \times 10^{-7}$
I3	Bxxx sxxx	2.0240	$9.7 \times 10^{-9}$	$5.9 \times 10^{-8}$
I4	Rxxxxx	0.2424	$5.0 \times 10^{-9}$	$3.0 \times 10^{-8}$
I5	Rxxxxx	0.2405	$4.3 \times 10^{-9}$	$2.6 \times 10^{-8}$
E1	Exxxx lxxxxx	1.2105	$9.1 \times 10^{-9}$	$5.6 \times 10^{-8}$
E2	Mxx	2.5030	$7.5 \times 10^{-9}$	$4.6 \times 10^{-8}$
E3	Mxx	0.9042	$6.0 \times 10^{-10}$	$3.9 \times 10^{-9}$
E4	Mxx	0.6832	$1.2 \times 10^{-8}$	$7.4 \times 10^{-8}$
E5	Cxxxxx	0.8476	$8.8 \times 10^{-9}$	$5.3 \times 10^{-8}$

Based on the table above, the Lifetime Excess Cancer Risk (LCR) results showed the values of LCR were in a range of  $10^{-9}$ - $10^{-7}$  due to the exposure towards Cr. It was exceeding the acceptable risk range of  $10^{-6}$ - $10^{-4}$  which indicates a clearly acceptable carcinogenic health risk.



Table 4.19 Carcinogenic health risk assessments for the exposure towards Chromium (Cr) concentration in the lipsticks sample based on the lipsticks brand (n=15).

Lipsticks code	Brand	Cr Mean		
		concentration (mg/kg)	LADD	LCR
C1	Sxxxx Gxxx	0.5077	$6.7 \times 10^{-8}$	$2.8 \times 10^{-6}$
C2	Sxxxx Gxxx	0.5778	$7.6 \times 10^{-8}$	$3.2 \times 10^{-6}$
C3	Sxxxx Gxxx	2.0610	$2.7 \times 10^{-7}$	$1.1 \times 10^{-5}$
C4	Mxxxxxxxx	1.4335	$1.9 \times 10^{-7}$	$7.9 \times 10^{-6}$
C5	Axxx	0.6234	$8.2 \times 10^{-8}$	$3.4 \times 10^{-6}$
I1	Sxxxxx sxxx	0.7956	$1.0 \times 10^{-7}$	$4.4 \times 10^{-6}$
I2	Lxxxxx pxxxx	0.4785	$6.3 \times 10^{-8}$	$2.6 \times 10^{-6}$
I3	Bxxx sxxx	2.0240	$2.7 \times 10^{-7}$	$1.1 \times 10^{-5}$
I4	Rxxxxx	0.2424	$3.2 \times 10^{-8}$	$1.3 \times 10^{-6}$
I5	Rxxxxx	0.2405	$1.1 \times 10^{-7}$	$1.3 \times 10^{-6}$
E1	Exxxx lxxxx	1.2105	$5.6 \times 10^{-7}$	$6.7 \times 10^{-6}$
E2	Mxx	2.5030	$1.2 \times 10^{-7}$	$1.4 \times 10^{-5}$
E3	Mxx	0.9042	$4.2 \times 10^{-7}$	$5.0 \times 10^{-6}$
E4	Mxx	0.6832	$3.1 \times 10^{-7}$	$3.8 \times 10^{-6}$
E5	Cxxxxx	0.8476	$3.9 \times 10^{-7}$	$4.7 \times 10^{-6}$

Based on the table above, the Lifetime Excess Cancer Risk (LCR) results showed the values of LCR were in a range of  $10^{-6}$ - $10^{-5}$  due to the exposure towards Cr. It was within the acceptable risk range of  $10^{-6}$ - $10^{-4}$  which indicates an acceptable carcinogenic health risk.

Table 4.20 Non-carcinogenic health risk assessments for the longest exposure (35 years) towards highest heavy metals concentration according to the lipsticks sample price categories (n=15).

Lipstick Category	HQ of Heavy Metals Exposure		
	Pb	Cd	Cr
I	$1.06 \times 10^{-6}$	$2.67 \times 10^{-7}$	$1.65 \times 10^{-6}$
II	$1.96 \times 10^{-6}$	$1.49 \times 10^{-7}$	$1.61 \times 10^{-6}$
III	$3.69 \times 10^{-6}$	$7.33 \times 10^{-7}$	$1.99 \times 10^{-6}$

Generally, The Hazard Quotient (HQ) value for worst case scenario of Pb , Cd. And Cr exposure towards the lipsticks consumer showed HQ less than 1 as the results on all lipsticks categories. It indicates a sign of clearly acceptable non-carcinogenic health risk.

Table 4.21 Carcinogenic chronic health risk assessments for the longest exposure (35 years) towards highest heavy metals concentration according to the lipsticks sample price categories (n=15).

Lipstick Category	LCR of Heavy Metals Exposure	
	Cd	Cr
I	$1.39 \times 10^{-6}$	$5.92 \times 10^{-5}$
II	$7.74 \times 10^{-7}$	$5.84 \times 10^{-5}$
III	$3.84 \times 10^{-7}$	$7.18 \times 10^{-5}$

Based on the table above, the Lifetime Excess Cancer Risk (LCR) results for the worst case scenario of exposure showed the values of LCR were in a range of  $10^{-7}$  to  $10^{-6}$  due to the exposure towards Cd. It was within the acceptable risk range of  $10^{-6}$ - $10^{-4}$  which indicates an acceptable carcinogenic health risk and clearly acceptable carcinogenic health risk. The LCR results for the worst case scenario of exposure towards Cr showed the values of  $10^{-5}$ . It indicates that an acceptable carcinogenic health risk even though the exposures were as high as 35 years long.

## CHAPTER 5

### 5. DISCUSSION

The number of selected lipsticks product as a sample was fifteen (n=15), 5 each for different price range categories which is cheap, intermediate, and expensive price range. The colors of the lipsticks sample for each of price categories were varied obtained from the results of respondent's questionnaire. The colors of pink, red and brown/natural color were existed in each of the price categories. An analytical estimation test of heavy metals concentration in the lipsticks samples (n=15) was performed for 3 elements which is lead (Pb), chromium (Cr), and Cadmium (Cd). The results of heavy metals concentration of Pb, Cr, and Cd in the lipsticks were obtained after the raw lipsticks sample (n=15) going through the sample preparation through microwave digestion and sample analysis using Inductively Coupled Plasma-Optical Emission Spectrometry (ICP-OES).

The recovery values obtained for the recovery sample confirmed the efficiency of the extraction method. The average recovery value of the recovery samples for chromium (Cr) was 91% calculated at 95% probability. The cadmium (Cd) average recovery values was 94% while extraction method for lead (Pb) had 77% average recovery value.

The blank sample was prepared and also being analysed before the other lipsticks samples were analysed using the ICP-OES. The calibration standards of 0.005 ppm, 0.05 ppm, 0.1 ppm, 0.5 ppm, 1.0 ppm, 3.0 ppm, and 5.0 ppm were injected to the ICP-OES instrument to obtain the calibration curve correlation. The range of calibration curve correlation ( $R^2$ ) obtained by using standard samples of Pb, Cr, and Cd known concentration were within 0.9994-0.9999. The calibration curve for Pb obtained by using standard samples of Pb known concentration was  $R^2=0.9995$ . The calibration curve of  $R^2=0.9998$  and  $R^2=0.9998$  were obtained for the heavy metals of Cd and Cr respectively. The calibration data are presented in Figure of 4.1, 4.2 and 4.3 respectively.

Based on the guidelines of Control of Cosmetic Products in Malaysia which is prepared in accordance to the ASEAN Cosmetic Directive, Pb, Cd, and Cr are included in the list of substances which must not form part of the composition of cosmetic products in Annex II-part 1 (NPCB, 2014). These heavy metals should not being added into the cosmetics during the manufacturing process as an ingredients formula. However, Pb, Cd, and Cr were found existed in all the lipsticks tested in this study. These existence could be due to the naturally occurrences of these heavy metals in the color additives and also could be due to the contamination in lipsticks manufacturing process. Naturally, each colour additive can have 20 ug/g of lead which higher in darker color and Galena (a color additive which contained lead sulphide naturally) can be added to shimmer or affixed to mica in lipstick (Ancient Egypt, 2013). While in manufacturing, the heavy metals sources might be come from

solder, leaded paints on manufacturing equipment, and also from lead-contaminated dust that existed in the manufacturing surroundings (Qureshi, 2011).

The results of Pb, Cd, and Cr concentrations were inserted in the SPSS software system to be analyzed. Descriptive statistical analysis of mean, median, minimum, maximum, and standard deviation of the mean concentration of the sample (N=15) were analyzed. The dependent variable which is the mean concentration of Pb, Cd, and Cr of the lipsticks samples (N=15) were analyzed according to the independent variable which is the price categories. The price categories were defined as “cheap” (category I-<RM29.99), “intermediate” (category II- RM30-RM59.99), and “expensive” (category III- >RM60).

Generally, the mean concentration of lead (Pb), cadmium (Cd), and chromium (Cr) on each lipsticks in category I, II, and III in Table 4.2, 4.3 and 4.4 had not exceeded the permissible limit value/references dose of IRIS EPA (2013) and Health Canada (2011) except for brown Mxx lipsticks in category III. A high concentration of Pb which is exceeding the Canada Health (2011) Pb concentration limit might cause a possibility to develop a chronic health risk which are the affects virtually in every system in the body such as the reproductive, neurological, hematopoietic, hepatic, and renal systems (Iman Al-Saleh, Al-Enazi, & Shinwari, 2009). Thus, the health risk assessments need to be done to estimates the possibility of developing these health risks since the limits set are supposed to provide a high level of protection to susceptible consumer (Al-Dayl *et al.*, 2011).

For Pb mean concentration according to the price categories in Table 4.5, the lipsticks in category III consisted of higher mean concentration compared to the other two categories. It is because of the impact of outliers Pb concentration in Mac brown lipsticks which contained highest concentration. A high Pb concentration in this lipstick might be due to the colour shade of the brown lipsticks which was recorded with high pigmentation based on the previous study (Piccinini *et al.*, 2013). The same condition occurred to the results of Sxxxx Gxxx brown lipstick which also had highest Pb concentration in Table 4.2. The previous study recorded that 33% of brown lipsticks contains higher Pb concentration compared to 30% pink lipsticks and 14% of red lipsticks (Piccinini *et al.*, 2013). A high Pb concentration also might be due to the non-intended presence of a small quantity of a prohibited substance, stemming from impurities of natural or synthetic ingredients, the manufacturing process, storage, migration from packaging, which is technically unavoidable in good manufacturing practice, can permitted provided that such presence is in conformity with article 3 stated in the article 17 of EC Reg. 1233/2009, which in turn requests the cosmetic products available on the market to be safe for human health when used under normal or reasonably foreseeable conditions of use.

Overall, statistical test for the Pb, Cd and Cr had shown non-significant correlation between the price categories and these heavy metals concentration level in the lipsticks sample. It had to be remembered that the small sample size of lipsticks for each of the price categories (N=5) might affects the results. However,

the analytical test of Kruskal-Wallis and Mann-Whitney U showed that the concentration Pb in price category I were statistically significant compared to the concentration of Pb in price category II. This finding can be correlated to the previous findings that the possibility of the lipsticks' price do influence the heavy metal concentration especially Pb in lipsticks (Piccinini *et al.*, 2013).

Further calculation of non-carcinogenic chronic health risk assessment for Hazard Quotient (HQ) showed the results that were less than 1 for all of the lipsticks in every price category. The results showed in Table 4.14 to 4.16 indicates that Pb, Cd, and Cr exposure through daily lipsticks consumption of 6.71 years mean exposure durations only caused the acceptable chronic health risk. Pb is widely known to be toxic to the reproductive system, the liver and kidney, and the immune system but evidence for lead as a carcinogen is still considered less clear (Silbergeld *et al.*, 2000). Thus, the Lifetime Excess Cancer Risk (LCR) were only done on Cd and Cr since the fact recorded from the previous study confirmed that these heavy metals did caused the carcinogenic health effects (Nawrot *et al.*, 2006; USEPA, 2013).

The table of 4.17 showed results of value in a range of  $10^{-9}$  to  $10^{-7}$  which indicates a "clearly acceptable" carcinogenic health risk for exposure towards Cd in lipsticks in mean exposure duration of 6.71 years. The LCR for Cr exposure in table 4.18 showed the results with value in a range of  $10^{-5}$  to  $10^{-6}$  indicate the "acceptable"

carcinogenic health risk towards the lipsticks consumer in mean exposure duration of 6.71 years.

However, the possibility of developing the non-carcinogenic health risk due to the exposure on these heavy metals might be varied according to the different length of exposure duration and concentration of the heavy metals. Thus, the HQ and LCR for the worst case scenario of the longest exposure duration with highest heavy metals concentration existed in the lipsticks were calculated to confirm this possibility.

Based on table 4.19, the possibility of developing the non-carcinogenic health risk due to the exposure on these heavy metals after counting in the worst scenario of exposure showed the values of HQ were below 1 indicates that the possibility of developing non-carcinogenic health risk was low.

Based on Table 4.20, the possibility of developing the carcinogenic health risk due to the exposure on these heavy metals after counting in the worst scenario of exposure showed the values of LCR were in a range of  $10^{-7}$  to  $10^{-6}$  due to the exposure towards Cd. It was within the “acceptable” risk range of  $10^{-6}$ - $10^{-4}$  which indicates an “acceptable” carcinogenic health risk and “clearly acceptable” carcinogenic health risk. The LCR results for the worst case scenario of exposure towards Cr showed the values of  $10^{-5}$ . It indicates that an “acceptable” carcinogenic health risk even though the exposures were as high as 35 years long.

## CONCLUSION

The lipstick samples (N=15) collected from the local markets in Malaysia were measured for the mean concentration of Pb, Cd, and Cr. Generally, the concentrations of these heavy metals in the lipsticks were not higher than the safe permissible limit of risk except the MAC brown lipsticks which containing high Pb concentration (15.44 mg/kg) when compared to Canada Health (2011) permissible limit. The Pb concentrations in lipsticks category I is significantly higher compare to lipsticks in category II. There is no significant non-carcinogenic and carcinogenic health risk effect due to the exposure on these heavy metals through lipstick consumption based on the health risk assessment in this study. Thus, the UPM female staffs who are the lipsticks consumer in UPM do possess low possibility of developing the chronic non-carcinogenic and carcinogenic health risk based on the results of this study.

## STUDY LIMITATIONS

The response rate of the respondents was less than 80 % due to the time constraint and the lack of human resources to approach them. Other than that, based on the observation throughout the survey, the person in-charge in every faculty in UPM was reflected not many female staffs applied lipsticks in their daily life. Even though there was a list of the UPM female staffs, the questionnaires were still hard to be distributed since the exact number of respondents that use lipsticks cannot be determined.

The small sample size could be contributed to the non significant association between the price categories of lipsticks and the lead, cadmium and chromium concentration in this study.

During the extraction of the heavy metals from lipsticks samples, nitric acid ( $\text{HNO}_3$ ) of 69% was used without any acid mixtures in the method of acid digestion. Previous study has reported the extraction of heavy metals using nitric acid ( $\text{HNO}_3$ ) and hydrofluoric acid (HF) mixtures in the acid digestion method has given a more efficient extraction by producing a clear extract without leaving any sediments of wax in the end of the acid digestions methods (Piccinini *et al.*, 2013). The types of digestion method and application of harsh acid solutions were known as efficient to improve the heavy metals recovery (Piccinini *et al.*, 2013).

## RECOMMENDATIONS

The survey need to be done on a suitable target population which having a characteristics of applying lipsticks as compulsory in their work routine. The examples of these target populations were those who involve with public service such as in marketing industry and entertainment industry.

There is a need to study the factor of color intensity associated to the heavy metals concentration in cosmetics since Piccini *et al.* (2013), Gondal *et al.* (2010), and Al-Saleh and Al-Enazi (2011) have reported the colour of the lipsticks could affect the heavy metals concentration. This statement is supported by the finding of this study that the brown lipsticks in price category I and III contained higher lead (Pb) concentration when compared to the pink and red lipsticks.

Previous study has reported Cd, Co, and Ni concentration in eye shadows were ranged from 1.00-33.04 ng/g, 0.70-161.0 ng/g, 15.00 -253.10 ng/g respectively (Volpe *et al.*, 2012). It is also suggested to study the heavy metals concentration in different type of cosmetics other than lipsticks such as eye shadow, blusher and face powder that sold in Malaysia local market. It is important to obtain the trend of heavy metals concentration in these cosmetics products and to estimates the health risk of these heavy metals exposure to them.

Increasing the sample size of cosmetics is deemed to improve the results of the statistical analysis. In study reported by Al-Saleh and Al-Enazi (2011), 72 lipsticks samples were analyzed to determine the lead concentrations in Arab. Besides that, the study of Piccinini *et al.*, (2013) has reported a significant association between different color lipsticks to the heavy metals concentration in 223 lipstick samples.



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## APPENDICES

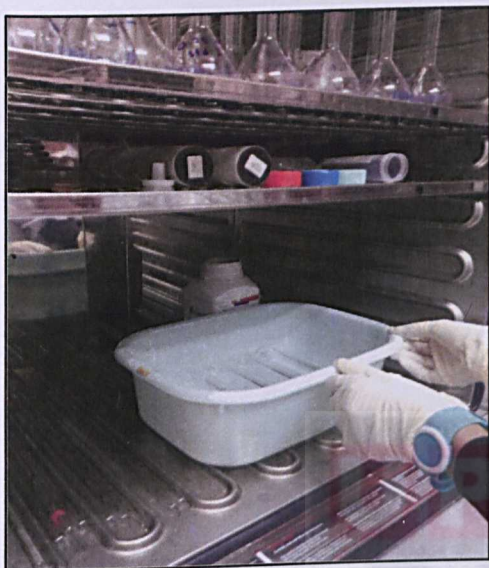


Figure 2 Vessel liners are inserted into the oven to be dried



Figure 1 Microwave vessel liners

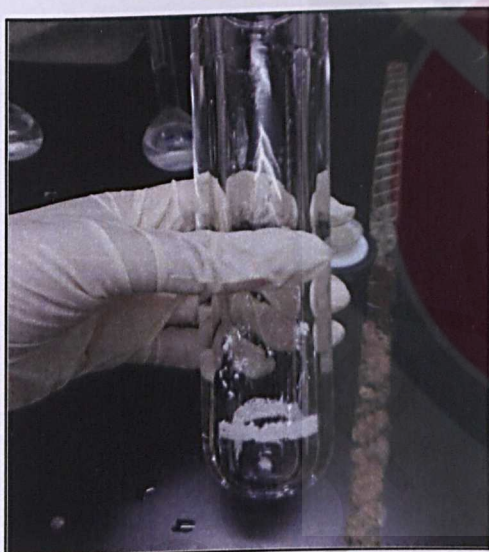


Figure 4 Sediments need to be filtered after the sample was digested



Figure 5 Estee lauder lipstick sample

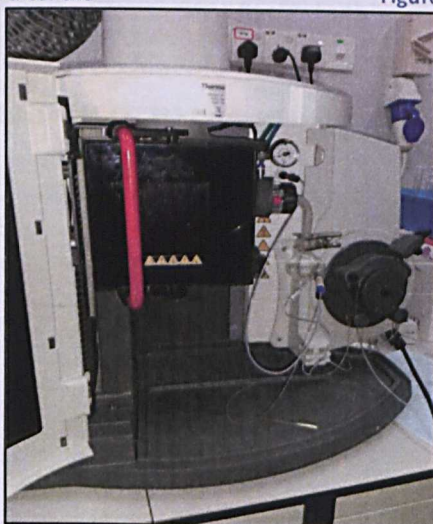


Figure 3 ICP-OES

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- Assoc. Prof. Dr. Azmawani Abd Rahman

b) Date of approval: 20/12/2013

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19 Februari 2014

Tuan,

**Permohonan Mendapatkan Jumlah Dan Senarai Nama Staf Wanita Yang Bekerja Di  
Universiti Putra Malaysia (UPM)**

Merujuk kepada perkara di atas, saya, Airin Zakaria, nombor matrik 156515, pelajar tahun akhir Bachelo Sains Kesihatan Persekitaran dan Pekerjaan ingin mendapatkan senarai nama dan jumlah staf sokongan dan staf akademik wanita yang bekerja di Universiti Putra Malaysia (UPM) untuk tujuan menjalankan Projek Tahun Akhir (EOH4999B) yang bertajuk "Kandungan Logam Berat Dalam Gincu dan Risiko Kesihatannya Terhadap Pengguna".

2. Untuk pengetahuan pihak tuan, senarai ini diperlukan untuk mengenal pasti bilangan staf sokongan dan staf akademik wanita di UPM yang menggunakan gincu dalam kehidupan seharian. Jumlah sebenar staf UPM ini akan digunakan untuk mengira jumlah responden yang diperlukan untuk tujuan soal selidik dalam projek tahun akhir ini.

3. Diharap pihak tuan dapat mempertimbangkan permohonan ini untuk melancarkan dan menayakan projek tahun akhir ini.

Sekian, terima kasih.

Yang Benar,

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18 FEBRUARI 2014

Tuan/Puan,

**Permohonan Mendapatkan Jumlah Dan Senarai Nama Staf Perempuan Fakulti Perubatan Dan Sains Kesihatan**

Merujuk kepada perkara di atas, saya, Airin Zakaria (156515), pelajar tahun akhir Bachelo Sains Kesihatan Persekitaran dan Pekerjaan ingin mendapatkan senarai nama dan jumlah staf sokongan dan staf akademik perempuan yang bekerja di Fakulti Perubatan dan Sains Kesihatan untuk tujuan menjalankan Projek Tahun Akhir (EOH4999B) yang bertajuk Penyelidikan Logam Berat Dalam Gincu dan Tahap Risiko Kesihatannya Terhadap Pengguna.

02. Untuk pengetahuan pihak tuan, senarai ini diperlukan untuk mengenalpasti pekerja perempuan Fakulti Perubatan dan Sains Kesihatan yang menggunakan gincu dalam kehidupan seharian. Selain itu, jumlah sebenar pekerja perempuan di Fakulti Perubatan dan Sains Kesihatan juga diperlukan untuk mendapatkan jumlah responden yang diperlukan dalam penyelidikan projek tahun akhir ini.

03. Diharap pihak tuan dapat mempertimbangkan permohonan ini untuk melancarkan dan mejayakan projek tahun akhir ini.

Sekian, terimakasih.

Yang Benar,

Airin binti Zakaria,  
Pelajar Tahun Akhir,  
B. Sc. Kesihatan Persekitaran dan Sains Kesihatan



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PENYELIDIKAN MELIBATKAN MANUSIA (JKEUPM)  
UNIVERSITI PUTRA MALAYSIA, 43400 UPM SERDANG,  
SELANGOR, MALAYSIA

## FORM B1: RESPONDENT'S INFORMATION SHEET AND CONSENT

Please read the following information carefully and do not hesitate to discuss any questions you may have with the researcher.

**1. STUDY TITLE :** Heavy Metals In Lipsticks And Its Associated Health Risk To Cosmetics Consumer.

**2. INTRODUCTION:** The main aim of this study is to determine the heavy metals concentration in lipsticks of a different range of price in the Malaysia market and to evaluate the potential health risk due to daily ingestion of lipsticks women who applied lipsticks on their lips. The questionnaire given is aim to determine the information on the most commonly used lipsticks among women. The importances to gain the information on the type of lipsticks that are commonly used are the researcher can identify the level of heavy metals in those lipsticks. The sociodemographic information such as weight and height of the respondents can also be gain from the questionnaire which is important to be included in the calculation of determining the risk of heavy metals exposure.

### 3. WHAT WILL YOU HAVE TO DO?

The respondents need to answer the questionnaire given within 15 minutes after been given. The researcher will collect the questionnaire after it is answered.

#### **4. WHO SHOULD NOT PARTICIPATE IN THE STUDY?**

The men and the women who are not applying lipsticks in their daily life

#### **5. WHAT WILL BE THE BENEFITS OF THE STUDY:**

**(a) TO YOU AS THE SUBJECT**

The respondent will received the summary of the study result on the level of concentration of heavy metals in lipsticks.

**(b) TO THE INVESTIGATOR**

The investigator will gain the information on the type of lipsticks that are commonly used so that the investigator can identify the level of heavy metals in those lipsticks. The sociodemographic information such as weight and height of the respondents can also be gain from the questionnaire which is important to be included in the formula of determining the risk of heavy metals exposure.

#### **6. WHAT ARE THE POSSIBLE RISKS?**

There's no possible risk in answering the questionnaire.

#### **7. WILL THE INFORMATION THAT YOU PROVIDE AND YOUR IDENTITY REMAIN CONFIDENTIAL?**

The respondent's information gained from the questionnaire will be remains confidential.

**8. WHO SHOULD YOU CONTACT IF YOU HAVE ADDITIONAL QUESTIONS DURING THE COURSE OF THE RESEARCH?**

The person that you may contact if you have additional questions during the course of the research is Miss Airin Zakaria (0138766078), the investigator in this research.



*Please initial here if you have read and understood the contents of this page \_\_\_\_\_*

**9. CONSENT**

I.....Identity Card  
No.....Address..... hereby  
voluntarily agree to take part in the research stated above \*(clinical /drug trial/video recording/  
focus group/interview-based/ questionnaire-based).

I have been informed about the nature of the research in terms of methodology, possible adverse effects and complications (as written in the Respondent's Information Sheet). I understand that I have the right to withdraw from this research at any time without giving any reason whatsoever. I also understand that this study is confidential and all information provided with regard to my identity will remain private and confidential.

I\* wish / do not wish to know the results related to my participation in the research

I agree/do not agree that the images/photos/video recordings/voice recordings related to me be used in any form of publication or presentation (if applicable)

\* delete where necessary

Signature:..... Signature:.....  
(Respondent) (Witness)

Date:..... Name :.....  
I/C No:.....

I confirm that I have explained to the respondent the nature and purpose of the above-mentioned research.

Date..... Signature:.....  
(Researcher)



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FACULTY OF MEDICINE AND HEALTH SCIENCES

**SECTION A: SOCIO-DEMOGRAFIC INFORMATION**

*Instruction: Please fill in the information below and tick ( / ) in a box given.*

1. Email address: \_\_\_\_\_

2. Age: \_\_\_\_\_ years old.

3. Race

<input type="checkbox"/>	Malay
<input type="checkbox"/>	Chinese
<input type="checkbox"/>	Indian
<input type="checkbox"/>	Others

4. Nationality

Malaysian

Foreign

5. Marital status

Married

Single

6. Occupation

UPM Staff

Lecturer

Student

7. Weight: \_\_\_\_\_ kg

8. Height: \_\_\_\_\_ cm

**SECTION B: THE LIPSTICKS USAGE**

1. State the lipsticks brand that you are usually use in your daily life

\_\_\_\_\_

2. Price range of the lipsticks

RM 10-29.90  RM30-59.90  RM 60-100

3. What do you think on your lipsticks price range

Cheap  Medium  Expensive

4. What is the lipsticks colour that you are usually use

Bright red  Brown  Pink

Other colour: \_\_\_\_\_

5. How long did you use lipsticks stated above in your daily life?

\_\_\_\_\_ years old

6. The frequency of you reapplying lipsticks in a day

1-2 times  3-4 times  5-6 times

7. The usual duration of you applying the lipsticks within a day

9 hour (office hour)  12 hour (half day)

Etc: \_\_\_\_\_ hour

Thank You for Your Cooperation



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## **BORANG B1: PENERANGAN DAN PERSETUJUAN RESPONDEN**

Sila baca maklumat berikut dengan teliti. Sekiranya anda mempunyai sebarang pertanyaan, sila kemukakan kepada penyelidik.

1. **TAJUK KAJIAN** : Logam Berat Dalam Gincu dan Risiko Kesihatannya Terhadap Pengguna Kosmetik

2. **PENGENALAN** : Tujuan utama kajian ini adalah untuk menentukan kepekatan logam berat dalam gincu yang berbeza kelas harga di pasaran Malaysia dan untuk menilai risiko kesihatan terhadap wanita yang menggunakan gincu di bibir mereka dalam kehidupan seharian. Borang soal selidik yang diberikan adalah bertujuan untuk menentukan maklumat mengenai gincu yang paling biasa digunakan di kalangan responden. Tujuan utama capaian maklumat mengenai jenis gincu yang biasa digunakan oleh wanita adalah untuk penyelidik dapat mengenal pasti tahap logam berat dalam gincu-gincu tersebut. Maklumat sosiodemografi seperti berat badan dan ketinggian daripada responden juga boleh menjadi mendapat daripada borang soal selidik yang penting untuk dimasukkan ke dalam pengiraan menentukan risiko pendedahan logam berat.

3. **APAKAH YANG PERLU ANDA LAKUKAN?**

Responden perlu menjawab borang soal selidik yang diberikan dalam tempoh 15 minit selepas diberikan. Penyelidik akan memungut borang tersebut selepas ia habis dijawab.

4. **SIAPA YANG TIDAK BOLEH MENYERTAI KAJIAN INI?**

Lelaki dan wanita yang tidak memakai gincu dalam kehidupan seharian mereka

## 5. APAKAH FAEDAH MENYERTAI KAJIAN INI?

### a) KEPADA ANDA SEBAGAI PESERTA?

Responden akan menerima ringkasan hasil kajian ke atas tahap kepekatan logam dalam gincu.

### b) KEPADA PENYELIDIK?

Penyelidik mendapat maklumat mengenai jenis gincu yang biasa digunakan supaya boleh mengenal pasti tahap logam dalam gincu yang selalu digunakan. Maklumat sosiodemografi seperti berat badan dan ketinggian responden juga boleh menjadi boleh diperolehi daripada soal selidik ini dan penting untuk dimasukkan ke dalam formula pengiraan dalam menentukan risiko pendedahan logam berat terhadap pengguna.

## 6. ADAKAH IA BERISIKO?

Tidak ada risiko yang mungkin berlaku dalam menjawab soal selidik.

## 7. ADAKAH MAKLUMAT DAN IDENTITI SAYA KEKAL RAHSIA?

Maklumat responden yang diperolehi daripada soal selidik ini akan kekal sulit.

## 8. SIAPA YANG SAYA PERLU HUBUNGI SEKIRANYA SAYA MEMPUNYAI SOALAN TAMBAHAN SEMASA MENGIKUTI PENYELIDIKAN INI?

Orang yang anda boleh hubungi jika anda ada soalan tambahan semasa kajian adalah Cik Airin Zakaria (0138766078), penyelidik dalam kajian ini.

*Sila tandatangan di sini sekiranya anda telah membaca dan memahami kandungan halaman ini*

---

## 9. PERSETUJUAN

Saya.....No ..... Kad  
Pengenalan.....beralamat.....  
.....dengan ini bersetuju untuk mengambil bahagian secara sukarela dalam penyelidikan yang tersebut di atas \*(kajian klinikal/percubaan ubat-ubatan/rakaman video/kumpulan sasaran/temuduga/ soal selidik).

Saya telah diberi penjelasan secara menyeluruh mengenai penyelidikan ini dari segi metodologi, risiko dan komplikasi (seperti tertulis pada Helaian Penerangan Responden). Saya memahami bahawa saya berhak menarik diri dari penyelidikan ini pada bila-bila masa tanpa memberi sebarang alasan.Saya juga memahami bahawa sebarang maklumat yang berkaitan identiti saya akan dirahsiakan.

Saya\* berminat / tidak berminat untuk mengetahui keputusan kajian yang melibatkan saya.

I setuju/tidak bersetuju untuk imei/gambar/rakaman video/ rakaman suara digunakan dalam apa jua bentuk penerbitan atau pembentangan. (sekiranya berkaitan).

\*potong yang tidak berkenaan

Tandatangan:..... Tandatangan:.....  
(Responden) (Saksi)

Tarikh:..... Nama : .....

No K/P:.....

Saya mengesahkan bahawa saya telah menerangkan kepada responden ini sifat dan tujuan penyelidikan yang tersebut di atas.

Tarikh  
Tandatangan:.....  
(Penyelidik)



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**BAHAGIAN A: MAKLUMAT Sosio-Demografi**

**Arahan: Sila isikan maklumat di bawah dan tandakan (/) dalam kotak yang diberikan.**

1. Alamat e-mel: \_\_\_\_\_

2. Umur: \_\_\_\_\_ tahun

3. Bangsa

<input type="checkbox"/>	Melayu
<input type="checkbox"/>	Cina
<input type="checkbox"/>	India
<input type="checkbox"/>	Lain-lain

4. Warganegara

Malaysia

Luar negara

5. Status perkahwinan

Bujang

Berkahwin

6. Pekerjaan

Staf UPM

Pensyarah

Pelajar

7. Berat: \_\_\_\_\_ kg

8. Tinggi: \_\_\_\_\_ cm

### BAHAGIAN B : PENGGUNAAN GINCU

1. Jenama gincu yang biasa anda gunakan dalam kehidupan harian anda

\_\_\_\_\_

2. Kadar harga gincu yang digunakan

RM 10-29,90

RM30 - 59.90

RM 60-100

3. Pandangan anda tentang kadar harga gincu tersebut

Murah

Sederhana

Mahal

4. Apakah warna gincu yang biasanya anda gunakan

Merah terang

Merah jambu

coklat

Warna lain : \_\_\_\_\_

4. Sudah berapa lama anda menggunakan gincu yang dinyatakan di atas dalam kehidupan harian anda ?

\_\_\_\_\_ tahun

5. Kekerapan anda menggunakan gincu dalam satu hari

1-2 kali

3-4 kali

5-6 kali

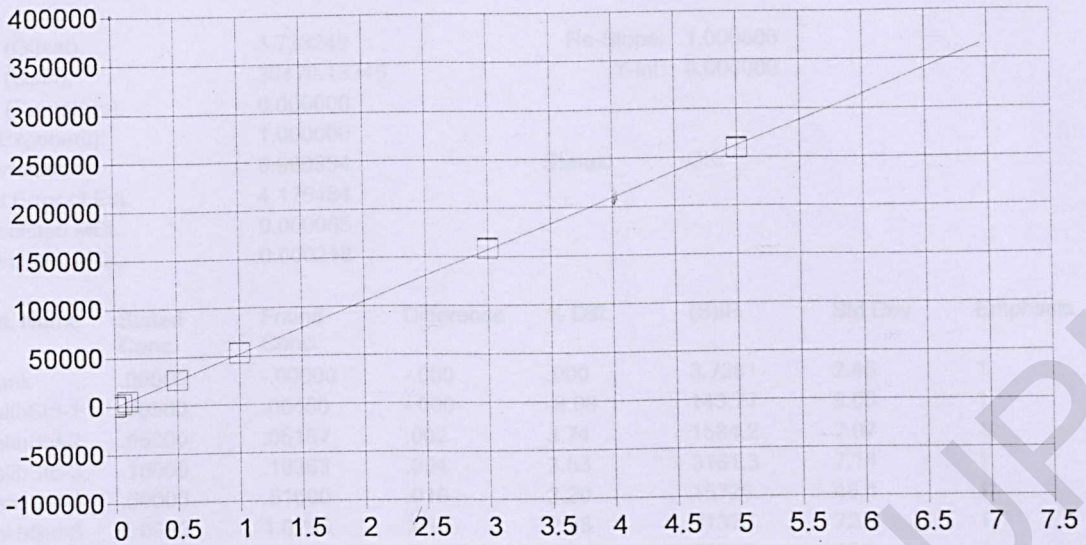
6. Tempoh biasa anda menggunakan gincu dalam masa sehari

9 jam (waktu pejabat)

12 jam (setengah hari)

Lain-lain : \_\_\_\_\_ jam

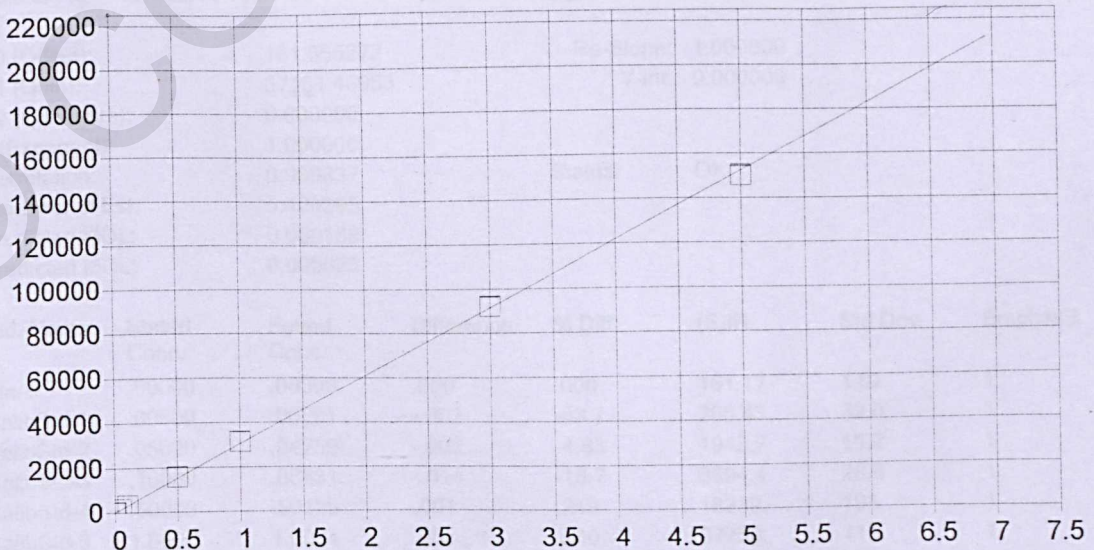
Terima kasih atas kerjasama anda



Date of Fit: 22/4/2014 10:57:50      Type of Fit: Linear      Weighting: 1/Conc

A0 (Offset): -2.227933      Re-Slope: 1.000000  
 A1 (Gain): 52419.49984      Y-int: 0.000000  
 A2 (Curvature): 0.000000  
 n (Exponent): 1.000000  
 Correlation: 0.999924      Status: OK.  
 Std Error of Est: 5.195647  
 Predicted MDL: 0.000036  
 Predicted MQL: 0.000121

Std. Name	Stated Conc.	Found Conc.	Difference	% Diff.	(S)IR	Std Dev	Emphasis
Blank	.00000	.00000	.000	.000	-2.2212	.540	1
CalibStd-1	.00500	.00450	-.001	-10.1	233.42	10.1	1
CalibStd-2	.05000	.05092	.001	1.85	2667.2	9.78	1
CalibStd-3	.10000	.10178	.002	1.78	5333.3	11.4	1
CalibStd-4	.50000	.51149	.011	2.30	26810.	4.01	1
CalibStd-5	1.0000	1.0201	.020	2.01	53469.	93.8	1
CalibStd-6	3.0000	3.0199	.020	.664	158300.	546.	1
CalibStd-7	5.0000	4.9463	-.054	-1.07	259280.	565.	1

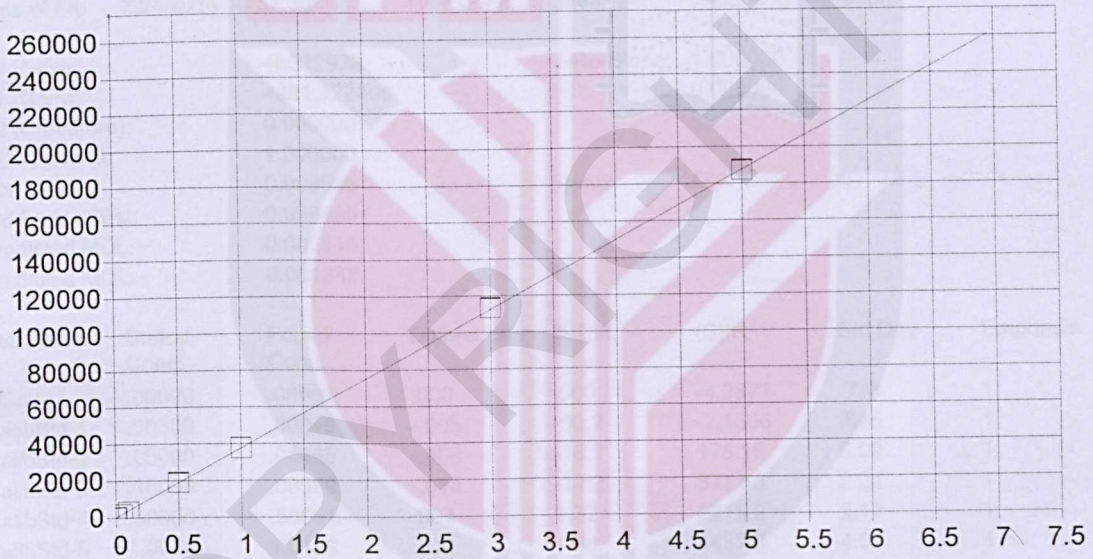


**Cd 228.802 {447}**

Date of Fit: 22/4/2014 10:57:50      Type of Fit: Linear      Weighting: 1/Conc

A0 (Offset): 3.733249      Re-Slope: 1.000000  
 A1 (Gain): 30470.13346      Y-int: 0.000000  
 A2 (Curvature): 0.000000  
 n (Exponent): 1.000000  
 Correlation: 0.999854      Status: OK.  
 Std Error of Est: 4.175484  
 Predicted MDL: 0.000065  
 Predicted MQL: 0.000218

Std. Name	Stated Conc.	Found Conc.	Difference	% Diff.	(S)IR	Std Dev	Emphasis
Blank	.00000	-.00000	-.000	.000	3.7261	2.46	1
CalibStd-1	.00500	.00460	-.000	-8.08	143.77	6.05	1
CalibStd-2	.05000	.05187	.002	3.74	1584.2	7.07	1
CalibStd-3	.10000	.10363	.004	3.63	3161.3	7.14	1
CalibStd-4	.50000	.51600	.016	3.20	15726.	46.1	1
CalibStd-5	1.0000	1.0295	.030	2.95	31373.	72.6	1
CalibStd-6	3.0000	3.0217	.022	.723	92075.	101.	1
CalibStd-7	5.0000	4.9277	-.072	-1.45	150150.	450.	1



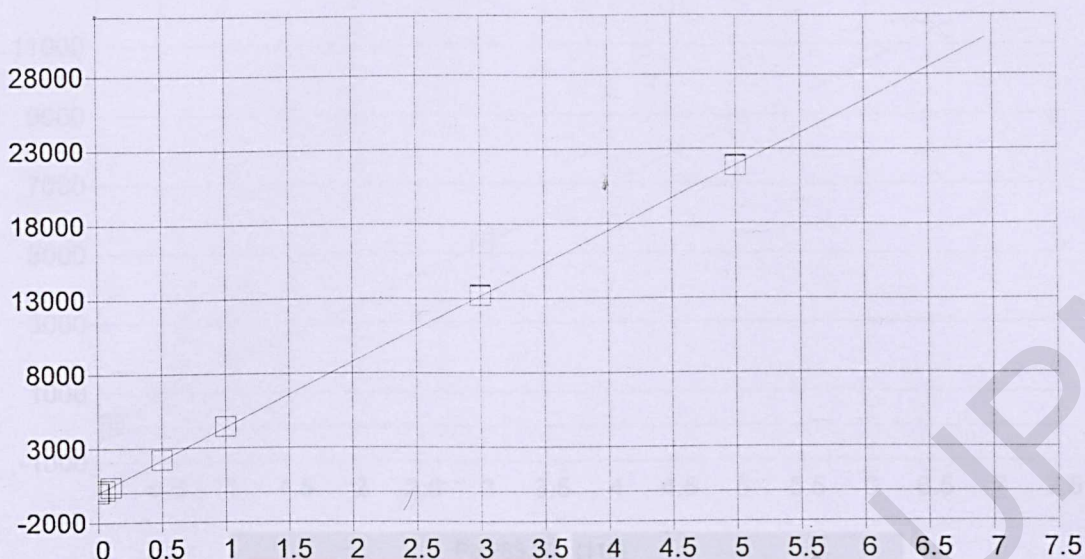
**Cr 283.563 {119}**

Date of Fit: 22/4/2014 10:57:50      Type of Fit: Linear      Weighting: 1/Conc

A0 (Offset): 161.055272      Re-Slope: 1.000000  
 A1 (Gain): 37461.46953      Y-int: 0.000000  
 A2 (Curvature): 0.000000  
 n (Exponent): 1.000000  
 Correlation: 0.999837      Status: OK.  
 Std Error of Est: 5.429295  
 Predicted MDL: 0.000188  
 Predicted MQL: 0.000626

Std. Name	Stated Conc.	Found Conc.	Difference	% Diff.	(S)IR	Std Dev	Emphasis
Blank	.00000	.00000	.000	.000	161.17	1.62	1
CalibStd-1	.00500	.00281	-.002	-43.7	266.43	32.0	1
CalibStd-2	.05000	.04759	-.002	-4.83	1943.7	19.2	1
CalibStd-3	.10000	.08631	-.014	-13.7	3394.4	28.6	1
CalibStd-4	.50000	.50125	.001	.249	18939.	195.	1
CalibStd-5	1.0000	1.0034	.003	.340	37750.	111.	1
CalibStd-6	3.0000	3.0228	.023	.761	113400.	400.	1

CalibStd-7 5.0000 4.9908 -.009 -.184 187120. 373. 1

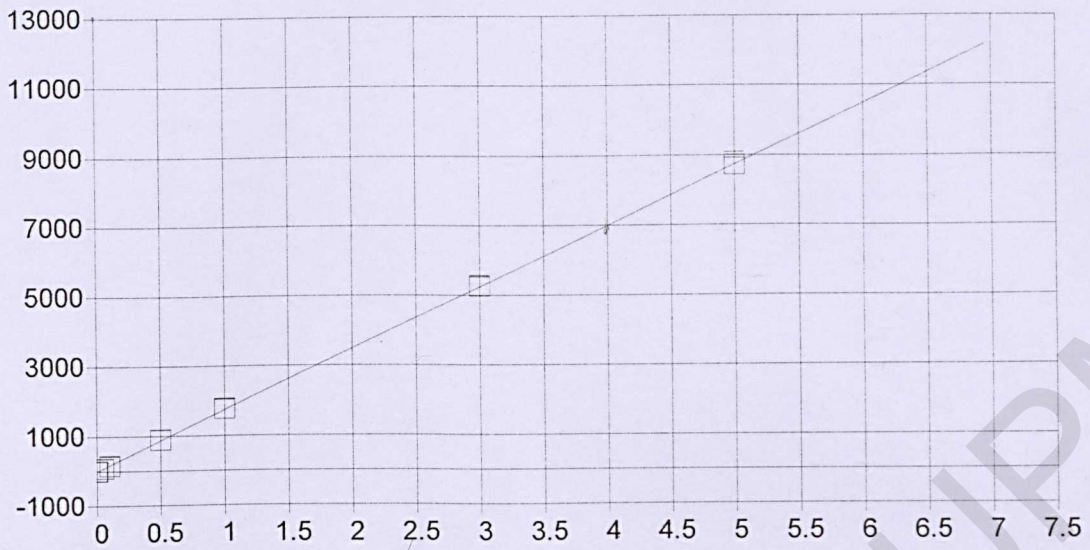


Pb 220.353 {453}

Date of Fit: 22/4/2014 10:57:50 Type of Fit: Linear Weighting: 1/Conc

A0 (Offset): -4.312938 Re-Slope: 1.000000  
 A1 (Gain): 4381.128400 Y-int: 0.000000  
 A2 (Curvature): 0.000000  
 n (Exponent): 1.000000  
 Correlation: 0.999595 Status: OK.  
 Std Error of Est: 0.999465  
 Predicted MDL: 0.000415  
 Predicted MQL: 0.001382

Std. Name	Stated Conc.	Found Conc.	Difference	% Diff.	(S)IR	Std Dev	Emphasis
Blank	.00000	.00001	.000	.000	-4.2871	.728	1
CalibStd-1	.00500	.00049	-.005	-90.2	-2.1596	.846	1
CalibStd-2	.05000	.04165	-.008	-16.7	178.15	6.09	1
CalibStd-3	.10000	.08580	-.014	-14.2	371.58	2.24	1
CalibStd-4	.50000	.50661	.007	1.32	2215.2	2.17	1
CalibStd-5	1.0000	1.0138	.014	1.38	4437.3	4.64	1
CalibStd-6	3.0000	3.0119	.012	.397	13191.	37.5	1
CalibStd-7	5.0000	4.9947	-.005	-.105	21878.	54.0	1



**Pb 283.306 {119}**

Date of Fit: 22/4/2014 10:57:50      Type of Fit: Linear      Weighting: 1/Conc

A0 (Offset): -3.927528      Re-Slope: 1.000000  
 A1 (Gain): 1747.455315      Y-int: 0.000000  
 A2 (Curvature): 0.000000  
 n (Exponent): 1.000000  
 Correlation: 0.999359      Status: OK.  
 Std Error of Est: 0.501729  
 Predicted MDL: 0.004442  
 Predicted MQL: 0.014805

Std. Name	Stated Conc.	Found Conc.	Difference	% Diff.	(S)IR	Std Dev	Emphasis
Blank	.00000	.00001	.000	.000	-3.9136	6.94	1
CalibStd-1	.00500	-.00174	-.007	-135.	-6.9712	4.15	1
CalibStd-2	.05000	.04245	-.008	-15.1	70.245	2.63	1
CalibStd-3	.10000	.08721	-.013	-12.8	148.46	6.35	1
CalibStd-4	.50000	.51057	.011	2.11	888.27	6.69	1
CalibStd-5	1.0000	1.0142	.014	1.42	1768.3	11.9	1
CalibStd-6	3.0000	3.0126	.013	.422	5260.5	25.2	1
CalibStd-7	5.0000	4.9897	-.010	-.206	8715.3	83.2	1