CHAPTER V

CONCLUSION

Dermatologists suggest to wear protective clothing, hats and sunglasses as well as apply sunscreens in order to protect the skin from the harmful effects of UV radiation (American Cancer Society, 2012). Sunscreens have been become to the top list of the cosmetic products used in daily life. However, the trends of sunscreen have been changed over time. From the survey for primary sunscreen products performed in local hypermarkets in Thailand during August to September, 2012, it was observed that those products were manufactured in many countries including USA, Australia, Poland, Taiwan and Thailand. The top three of UV filters observed among thirty-three sunscreen products were EHMC, titanium dioxide and BMDBM, respectively. BZ-4, DAHB, DTS, EHT and TDSA were not widely used UV filters. Interestingly, the results of this survey were similar to those observed by Kerr (2010) who surveyed the UV filters used in 337 sunscreen products in Dundee, UK, in 2010. Moreover, the UV filters observed in Thailand according to this study were in the top rank of UV filters observed in Dundee, UK. In addition, the combination of between two to eight UV filters was observed in most commercial products and there was no single used of UV filter in sunscreen products. The reason may be due to the combination of more than one UV filter provides the absorption in different region of UV radiations and thus results in a high and broad UV protection efficacy. However, as the high concentration may cause many adverse effects. Therefore, the international regulatory agencies including Thai FDA establish lists of approved UV filters with the maximum concentration allowed to be used in sunscreen. However, an official analytical method is not officially available for determination of UV filters. Thus, the aim of this thesis was to develop a HPLC method for simultaneous determination of UV filters commonly used in sunscreen products in a single analysis. The ten UV filters which were selected to be assayed in this study were BZ-3, BEMT, BMDBM, ED-PABA, EHS, EHT, HMS, MBBT, OCR and EHMC. The HPLC method was developed by using stationary phase C18 (250 x 4.6 mm, 5 μm) with 20μL of injection volume, and UV detected at 325 nm. The

separation was performed under an isocratic and gradient mode. An isocratic method is simple and convenient. However, it may not suitable for a separation of complex substances. Although the gradient mode is more complicated, it also provides a beneficial for a separation of complex substances which have significant difference in polarity properties. Finally, the optimal condition which provided a resolution in acceptable range ($R_S \geq 1.5$) by using a gradient mode was complying with the continuous change of acetonitrile: ethyl acetate: water (94:4:2, v/v/v) at 0-3 min and acetonitrile: methanol (50:50, v/v) at 8.1-15 min. Total analysis time required was 16 min. The method was validated following ICH Q2 (R1) guideline in order to certify the validity and reliability in the analysis of the developed method. The results showed the good correlation in calibration curve with the correlation coefficient more than 0.999 and the accuracy showed in percentage of recovery 96-104%. Limit of detection and limit of quantitation were 0.05 μg/mL and 5 μg/mL, respectively. The method was successfully applied for the analysis of UV filters in sunscreen samples. The sunscreen samples, including standard sunscreens, in-house formulated sunscreens and commercial sunscreens, were examined to confirm the applicability of the method. It was shown that the method could correctly analyzed the amount of UV filters containing in the standard and formulated sunscreen formulations. For commercial sunscreens, the UV filters observed from HPLC analysis conformed to the list of ingredients which shown on the product labeling. The percentage of UV filters analyzed in all commercial products was within the permitted authorized levels regulated by Thai FDA. The analysis of developed method was not interfered by ingredients of base formulation such as emulsifiers (e.g. glyceryl monostearate), oils (e.g. isopropyl palmitate, jojoba oil), polymers (e.g. acrylates/C10-30 alkyl acrylate cross-polymer) preservatives (e.g. methyl paraben, propyl paraben, phenoxyethanol) and additives (e.g. vitamin C, vitamin E, niacinamide). On top of that, physical UV filters including titanium dioxide and zinc oxide did not interfere with the analysis of these ten chemical UV filters. The study achieved the objective which proposed to survey the UV filters commonly used in sunscreen products in order to obtain updated information on the usage of UV filters and to develop an efficient, fast and economical HPLC method for simultaneous determination of UV filters frequently found in commercial sunscreen products. As describe above, the results of this survey found that the UV filters observed in Thailand

were in the top rank of the UV filters which observed in the United Kingdom by Kerr (Kerr, 2010). Therefore, the developed method is not only able to analyze the ten UV filters in commercial sunscreens of Thailand, but it is also able to analyze these ten UV filters in commercial sunscreens of UK and worldwide. The method provides a beneficial to the regulatory authorities as well as the cosmetic companies for inspection, quality control and determining of photostability of commercial sunscreen products.

Suggestions arising from the study

1. In this study, a chemical filters named diethylaminohydroxybenzoyl hexylbenzoate (DAHB) was found to overlap with the peak of OCR and thus it is not included in this study. However, if a sunscreen product does not consist of OCR, this developed method can be applied to analyze DAHB.

2. Although the gradient HPLC method is more effective to simultaneously analyze various UV filters in single injection, it is more complex and expensive to implement compared with isocratic method. Therefore, if a manufacturing aims to analyze UV filters detected in the first group of chromatogram in isocratic HPLC analysis (i.e. BZ-3, OCR, BMDBM, EHMC, ED-PABA, EHS and HMS), the isocratic method (A condition of acetonitrile: ethyl acetate: water (95:5:2, v/v/v), flow rate 1.0 mL/min, injection volume 20 µL, reversed phase column C18 was used with a temperature control at 25 °C and detected at 325 nm) is recommended to be applied for routine analysis in order to save costs.