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Summary of PhD thesis

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## Lipid oxidation in skincare products

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## Summary

Skincare products are functional products containing multiple ingredients that serve various purposes in the formulation. One of the main ingredients in skincare products are lipids. Traditionally, most of the lipids added are saturated lipids to make them less vulnerable to lipid oxidation. However, recent studies have shown that unsaturated lipids improve skin appearance and function more than saturated lipids. Unsaturated lipids are more susceptible to lipid oxidation than saturated lipids. Therefore, unsaturated lipids and use of multiple ingredients challenge understanding and predicting lipid oxidation in the skincare product. Moreover, volatile lipid oxidation products can affect product odour in concentrations below 1 ng/g for some volatile compounds.

The objective of the present thesis was to investigate lipid oxidation in skincare products. Firstly, the extent of lipid oxidation was examined in skincare products, by analysing primary and secondary volatile oxidation products. In addition, sensory evaluation was conducted to examine the odour changes that appeared. The progress of lipid oxidation in all three analyses was compared to find markers for lipid oxidation. Moreover, the complex reactions behind the formation of volatile oxidation products were investigated in detail for the volatile compounds that increased the most. Evaluation of the physical and chemical including oxidative stability can be a bottleneck in product development. To combat this bottleneck, several approaches that can initiate lipid oxidation were examined with the aim to be able to predict oxidative stability faster.

The results showed that two volatile aldehydes, pentanal and heptanal, were good markers for monitoring progress of lipid oxidation in prototype lip care formulations (lip balm), prototype cleansing formulations and facial moisturiser prototypes with seaweed antioxidants added. However, only pentanal could be used as a marker in studies of the prototype skin cream formulation because heptanal was low and stable even though other oxidation products increased. Therefore, pentanal was a universal marker for lipid oxidation in all skincare products used throughout this thesis. Three types of seaweed extracts derived from brown alga, *Fucus vesiculosus*, were added as antioxidants to facial moisturiser prototype to improve oxidative stability. The oxidative stability under photo-activated oxidation was increased most by *Fucus vesiculosus* water extract. The *Fucus vesiculosus* water extract may decrease photooxidation due to a protective effect of the carotenoids present in the extract. However, *Fucus vesiculosus* acetone extract was more

efficient in preventing hydroperoxide decomposition to volatile compounds than the water extract. Furthermore, the *Fucus vesiculosus* acetone extract protected best against thermo-activated oxidation.

The effect of lipid composition on lipid oxidation was evaluated in prototype cleansing and lip care formulations. The prototype cleansing and lip care formulations had lipid content at 8 % and 36 %, respectively. The prototype lip care formulations with the highest lipid content also had the lowest oxidative stability. Thereby, the oxidative stability improved when the lipid content was reduced. However, minor changes in the lipid composition between two prototype cleansing formulation, and storage at 2°C did not show any clear difference. Nevertheless, storage at 20°C with exposure to light gave rise to a significantly higher concentration of heptanal in prototype cleansing formulation 1 than in formulation 2 after 56 days of storage. Furthermore, the intensities of the sensory attributes also increased with increased amount of unsaturated fatty acids.

The effect of storage conditions on lipid oxidation was evaluated by storing skincare products at temperatures ranging from 2°C to 50°C. Overall, a non-linear relationship was observed between the concentration of volatile compounds and temperature. In addition to temperature, the effect of exposure to light was also investigated. Exposure to light resulted in equal or an increased peroxide value and concentration of volatile oxidation products. A sensory profiling showed a clear effect of increased temperature on the sensory attribute intensities in the prototype cleansing formulation. Antioxidant addition resulted in less sensitivity towards photooxidation, a lower concentration of volatile compounds and intensities of attributes. Attributes that increased in sensitivity were suggested by partial least squares regression to be linked to formation of pentanal and butane nitrile. However, addition of pentanal and butane nitrile to prototype cleansing formulation did not result in the suggested attributes, probably because a combination of several volatile compounds gave rise to the attributes.

Butanal and pentanal were initially present or appeared in most lipid containing raw materials. One raw material, isoamyl p-methoxycinnamate, had a high concentration of 3-methyl-1-butanol. The main reaction route leading to the formation of 3-methyl-1-butanol may be direct hydrolysis of the ester-group. Butane nitrile did not appear in any raw materials, but it may be a by-product from azobisisobutyronitrile decomposition or a migrant from the plast packaging material. Lastly, approaches to predict oxidative stability faster were examined. Three initiators were able to accelerate lipid oxidation. However, only  $\text{FeCl}_2/\text{H}_2\text{O}_2$  was able to make a fast and correct prediction

of the oxidative stability in both prototype skin cream formulations and facial moisturiser prototypes containing *Fucus vesiculosus* extracts.

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