Online Profiling of Oxidation Byproducts in Capsuled Fish Oil

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The long-chain, unsaturated fatty acids abundant in fish oil products are known to be extremely susceptible to oxidation processes. This work demonstrates use of a TOFWERK Vocus PTR-TOF for on-line monitoring of volatile oxidation byproducts in capsuled fish oil. This method, which requires no sample preparation or pretreatment, can be used to non-invasively assess fish oil degradation levels during production processes or for high-throughput characterization of product samples.

Two sets of measurements were performed. In the first, fish oil was removed from capsules, placed in temperature regulated vials (30°C), and exposed to high concentrations of ozone to simulate atmospheric oxidation. The headspace of the vials was continuously sampled by a Vocus PTR-TOF to monitor the rapid evolution of oxidation products. In the second experiment, an equal amount of capsule fish oil was placed inside a new, transparent bottle every day for six consecutive days and sealed at room temperature under ambient air. After day 6, the headspace of each of the six bottles was measured by the Vocus PTR-TOF to characterize oxidation and aging products as a function of time.

Figure 1 profiles the rapid formation of selected volatile oxidation byproducts in the headspace of capsuled fish oil in the minutes following ozone injection. Most of the oxidation products emerged seconds after ozone infusion.

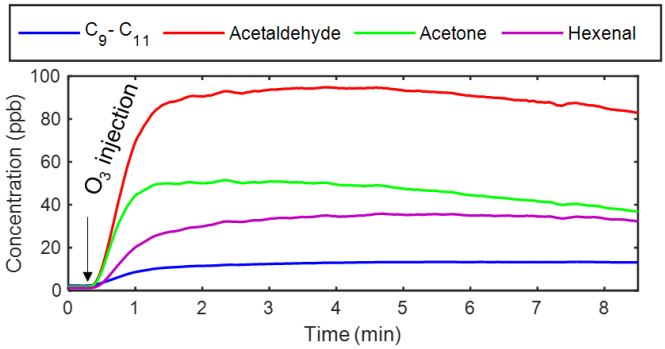

Figure 1 Continuous measurement of oxidation byproducts in the headspace of fish oil before and after ozone treatment. *Most of the oxidation products emerged seconds after ozone infusion.*

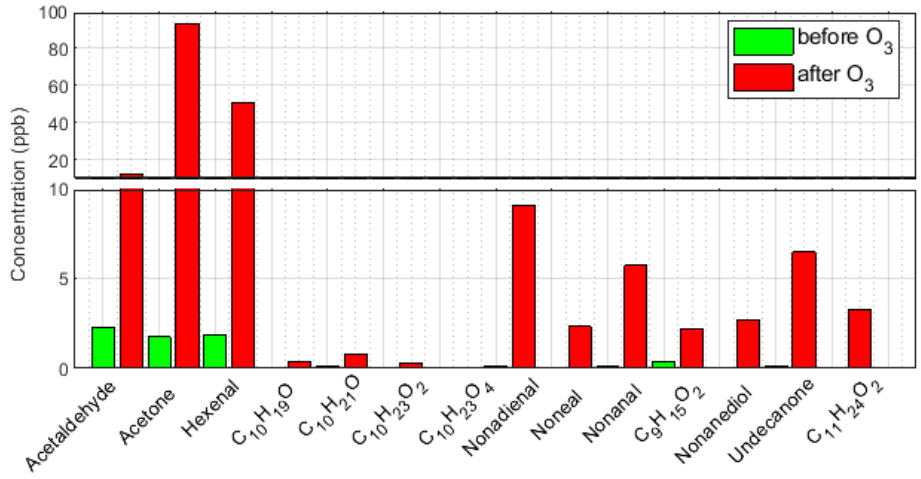

Figure 2 Concentration of selected oxidation byproducts measured in the headspace of fish oil before and after ozone treatment. *The oxidation produced many high molecular weight functionalized compounds that can serve as unique traces for diagnosing fish oil degradation.*

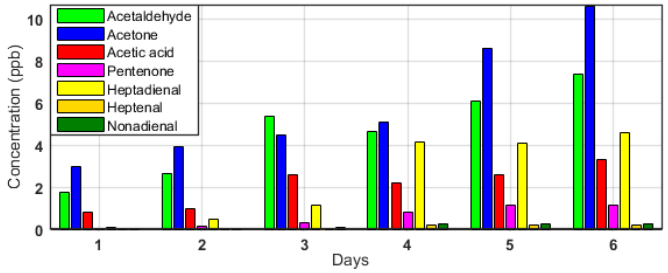

Figure 3 Concentration of oxidation byproducts in the headspace of fish oil stored under closed ambient air for an increasing number of days. *The concentrations of all species are positively correlated with the aging time but plateau at different rates.*

Figure 2 compares the concentrations of a broader range of oxidation byproducts in the fish oil headspace before and after the ozone treatment. The concentration of acetaldehyde and acetone (resp-propanal) increased by factors of ~8 and ~50, respectively. Other major peaks dominating the O_3-treated mass spectra were assigned as the known degradation byproducts hexenal, propanediol and butanal.

Additionally, the ozone introduction produced enormous increases in large oxygenated species, such as nonenal, nonanal, nonanedione. In comparison to the common VOCs which may originate from various sources, these higher molecular weight and more functionalized molecules can serve as unique tracers for diagnosing the degree of fish oil degradation.

Figure 3 shows the concentration of selected volatile species from samples aged under ambient atmosphere for increasing numbers of days. The concentrations of all species are positively correlated with the aging time. Some VOCs plateaued earlier than others, likely due to various degradation pathway involved. Different external stimulus such as radiation, ozone,

temperature and humidity are believed to heavily influence the headspace composition of aged samples. A systematic study involving all experimental factors could likely pinpoint qualitative and quantitative VOC markers for oxidation of unsaturated fatty acids in fish oil.

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