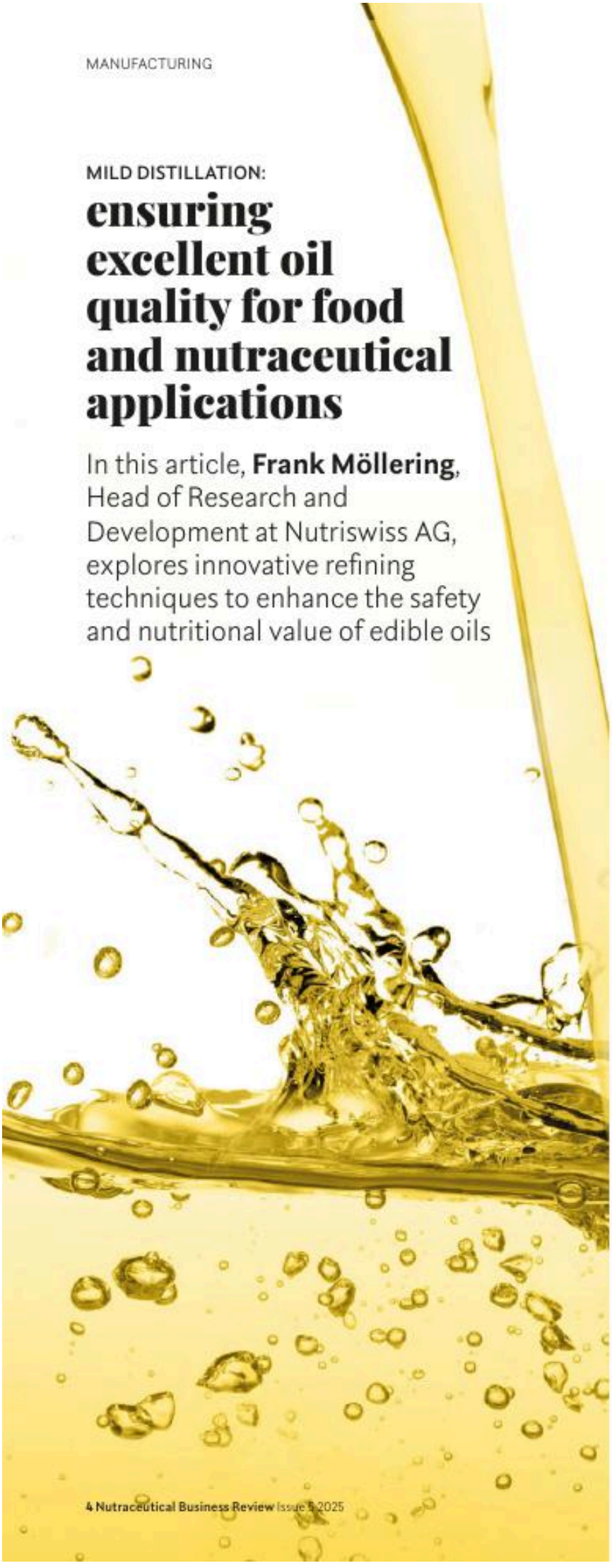
MILD DISTILLATION:

ensuring excellent oil quality for food and nutraceutical applications

In this article, **Frank Möllering**, Head of Research and Development at Nutriswiss AG, explores innovative refining techniques to enhance the safety and nutritional value of edible oils



Following the publication of Jan Velíšek's work on 3-chloropropane-1,2-diol (3-MCPD) levels in edible oils, the research and development team at Nutriswiss AG has been working to find ways to avoid process contamination during refining.[1] The focus was on infant formulas as a particularly sensitive product group. Many formulations were found to comprise — and still contain — palm oil, which has a high capacity to form 3-MCPD and glycidol.

Many strategies have been used to develop new processes and, undoubtedly, the best approach is to prevent the formation of contaminants with optimised harvesting and rapid processing at the source. In this way, levels of <500 µg/kg of 3-MCPD can be achieved. The disadvantage of this strategy is its dependence on selected suppliers. Irrespective of the quality of the raw materials, Nutriswiss has therefore developed a way to meet the need for particularly low 3-MCPD levels in infant formulas.

The focus is not just on 3-MCPD; the presence of genotoxic esters also needs to be controlled as they make refining seed oils such as sunflower, soya or maize germ a technological challenge. In addition, contaminants such as pesticides from non-organic cultivation or polycyclic aromatic hydrocarbons (PAHs) can also be found in conventional or even organic oils.

Short-path distillation: a key component of process control

If the glycidol content is to be kept as low as possible (<50 µg/kg) and low temperatures are used during deodorisation, the presence of certain impurities cannot be completely mitigated. A reliable and sophisticated process to solve this problem is short-path or molecular distillation (SPD). It can be used to remove contaminants from fats and oils very efficiently or to significantly reduce their levels. However, it is important that the product quality remains stable or is improved compared with conventional processes. Nutriswiss has done extensive tests to determine the ideal process parameters to achieve this goal.

The SPD used by Nutriswiss AG is a continuous vacuum distillation process supplied by VTA Verfahrenstechnische Anlagen GmbH & Co. KG (Figure 1). When in use, a scraper or wiper inside the cylindrical evaporator distributes the oil to be distilled in a thin layer on the heated wall of the

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evaporator. Because the apparatus operates at a vacuum of up to 10^{-1} mbar, volatile components such as pesticides and free fatty acids, as well as tocopherols, evaporate more easily under low thermal stress. They are subsequently liquefied again and removed via the nearby condenser; the cleaned oil then flows off the evaporator wall. The evaporation rate is normally in the range of a few per cent; the temperature and pressure depend on the oil to be processed and the substances to be separated.

Customising the refining strategy

Depending on the quality of the starting oil, Nutriswiss uses an individual combination of pretreatment, SPD and mild deodorisation. The process conditions for SPD are selected in such a way that the beneficial ingredients of the oils, especially the tocopherols, are maintained to the same extent as with classical refining.

In addition, internal studies have optimised the refining process so that the tocopherols in the base oil are largely retained or even increased. This can be explained by the cleavage of dimeric bonds between tocopherol molecules or ester bonds between tocopherols and other compounds. With an appropriately pretreated seed oil and process parameters chosen to maintain a target tocopherol content (comparable with physical refining), the following reductions can be expected for typical pesticides: anthraquinone – >92%; biphenyl – >97%; piperonyl butoxide – >90%; pirimiphos-methyl – >80%; and folpet – >93%.

Heavy PAHs can also be reduced by approximately 95% and activated carbon dosing can be reduced or even eliminated. For comparison, the mild refining process with deodorisation temperatures <200 °C is shown in Figure 2. For all the pesticides mentioned, the levels after SPD treatment were below the detection limit.

Short-path distillation reduces hydrocarbon contamination

The EU's proposed limit of 2 mg/kg for oils and fats continues to be criticised by NGOs and values of <1 mg/kg (detection

Figure 1: Industrial SPD plant supplied to Nutriswiss AG by VTA Verfahrenstechnische Anlagen GmbH & Co. KG (Copyright: VTA)

Figure 2: Pesticide levels are significantly lower after SPD treatment (Copyright: Nutriswiss)

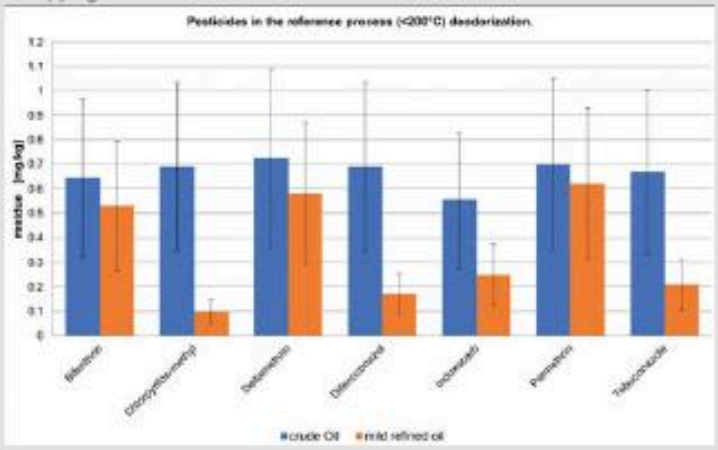
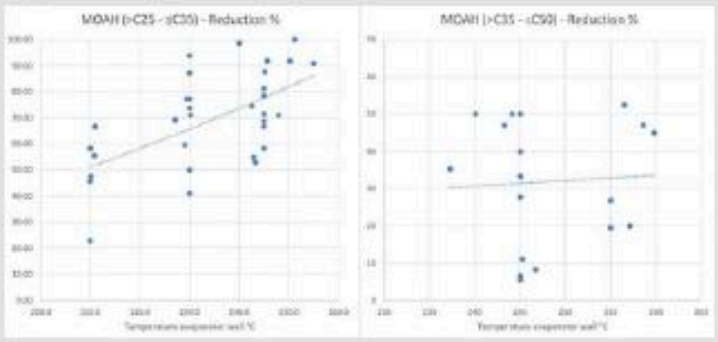


Figure 3: The MOAH fraction >C25 to ≤C35 can be expected to be reduced by about 70% and the fraction >C35 to ≤C50 by approximately 30% (Copyright: Nutriswiss)



limit) are required for mineral oil aromatic hydrocarbons (MOAHs). Even under the guidelines described above to preserve tocopherols while avoiding the formation of 3-MCPD/glycidol, it is possible to effectively reduce both mineral oil saturated hydrocarbons (MOSHs) and MOAHs using the SPD process.

However, the overall reduction rates are strongly dependent on the distribution of the individual mineral oil hydrocarbon (MOH) fractions. For example, the MOAH fraction >C25 to ≤C35 can be expected to be reduced by about 70% and the fraction >C35 to ≤C50 by approximately 30% (Figure 3).

Other positive effects of short-path distillation

Although the use of mild refining (steam temperature <200 °C) does not significantly reduce diethylhexyl phthalate (DEHP), another contaminant that can enter edible oils from plastic products as a plasticiser, SPD does have a good reduction effect on orthophthalates. The lowering of DEHP in crude hazelnut oil has previously been described.[2] With the process parameters typically used for seed oils, reduction rates of approximately 95% can be expected for DEHP (Figure 4). For DEHT, it is approximately 80%.

Another side-effect of SPD can be seen in the following example: palm oil fractions are often purchased by manufacturers as so-called RBD (refined, bleached and deodorised) fats. In principle, these have a fixed 3-MCPD content. Owing to the effects of transport and storage, however, these fats can no longer be considered to be of refined quality and require post-processing. An internal study was done at Nutriswiss to assess the influence of SPD on product stability as a result of post-refining, which yielded positive effects (Table I).

Mild refining aspects in combination with SPD

Physical refining has been used for the last 30–40 years. It ensured that a certain amount of impurities were removed in addition to the reduction of free fatty acids. However, it is known that this type of refining introduces process contaminants such as 3-MCPD/glycidol and trans fatty acids. Mild refining with the alkaline neutralisation of free fatty acids and subsequent deodorisation at <200 °C avoids the formation of these process contaminants and also results in more stable products (in terms of shelf-life and sensory properties).

Based on practical experience, Nutriswiss has been combining mild refining with SPD for several years to maximise the benefits of both processes. The company is currently working with a German university to test the effect on the quality of fatty acid oxidation products such as E,E-hydroperoxy, E,E-hydroxy, trans-epoxy and erythro-dihydroxy fatty acids. Initial results show that the combination of mild refining and SPD does not result in any significant changes compared with mild refining alone. In summary, the combination of mild refining and SPD results in a product of equivalent quality and, in some cases, one that is even more stable than obtained from mild refining alone. In addition, the purity of such a product is significantly higher and can even exceed the results of typical physical refining with its associated high temperatures.

References

1. www.researchgate.net/publication/6678913_Fatty_acid_esters_of_3-chloropropane-12-diol_in_edible_oils
2. https://onlinelibrary.wiley.com/doi/abs/10.1002/ejlt.201600001.

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Table I: Effects of SPD on storage stability compared with conventional post-refining

	RBD palm stearin	Post-refining RBD palm stearin incl. SPD	Post-refining RBD palm stearin
3-MCPD [mg/kg]	1.02	0.68	1.13
Glycidol [mg/kg]	0.39	0.04	0.02

Figure 4: With process parameters typically used for seed oils, reduction rates of around 95% can be expected for DEHP (Copyright: Nutriswiss)