Review on palm oil contaminants related to 3-monochloropropane-1,2-diol (3-MCPD) and glycidyl esters (GE)

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Abstract

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1. Introduction

After independence in 1957, agriculture played a significant role to bridge the gap between rural poor and urban area. Today's oil palm industry has become one of the key contributors to Malaysia's gross domestic product (GDP) around 5 - 7% (Nambiappan *et al.*, 2018). However, the oil palm industry does not always go well as it cannot avoid facing critique against issues such as environment, social, and health. Even though the negative campaign is not new, but current action led by the west influenced the oil palm economic performance in Malaysia.

3-Monochloropropanediol (MCPD) esters and glycidyl esters (GE) are categorized as food process contaminants, which are formed during the refining of palm oil mainly in the deodorization step. In 2016, the European Food Safety Authority (EFSA) published a report on the risk of 3-MCPD esters on human health which concluded that the free form and bound fatty acid esters of 3-MCPD caused kidney failure (EFSA, 2016). Realizing the fact that this issue must be well addressed, many researchers perform rigorous studies related to cause and mitigation methods that could be done to make sure the concentration of 3-MCPD within the allowable limit. The tolerable daily intake (TDI) for 3-MCPD is 0.8 µg/kg as derived by the EFSA. Meanwhile, for the GE, T25 value (representing the dose corresponding to a 25%

The issue of food safety is a major concern in the oil palm industry as it will affect national income. 3-MCPD and GE are contaminants formed during the refining of palm oil, mainly in the deodorization step. Palm oil was reported to contain one of the highest levels of these contaminants amongst all vegetable oils. Both 3-MCPD and GE are characterized as a possible risk to human health. This review aimed to provide a comprehensive summary of the 3-MCPD and GE precursors and mitigation strategies to minimize the 3-MCPD and GE formation. Therefore, the oil palm industry should address these issues and find ways to comply with food laws, acts, regulations, and standards enforced by local and international authorities.

incidence of tumours) of 10.2 mg/kg bw per day for the incidence of tumours observed in rats. The panel did not set any safe level since there is sufficient evidence that GE is genotoxic and carcinogenic. In refined, bleached, and deodorized (RBD) palm oil, the amount of 3-MCPD esters between 0.2 and 4 mg/kg due to different raw oil quality and processing conditions (Crews *et al.*, 2013).

Several approaches have been taken by many researchers to reduce 3-MCPD and GE in refined oil at various stages of oil production. This paper is not only to review different types of mitigation strategies to minimize 3-MCPD and GE but also to organize them into appropriate orders from the beginning, starting from the field to the milling stage up to the final stage, which is the refinement.

2. 3-MCPD and GE precursors

Prior to the investigation on how to mitigate the formation of 3-MCPD and GE, it is important to gain some knowledge of their precursors and their formation pathways. By knowing so, the researcher can reduce or eliminate the precursors. The MCPD fatty acid esters formed when the mono-, di- and triacylglycerols (MAG, DAG, TAG) accept chloride from chlorin donor substance under high temperature (Destaillats *et al.*, 2012a; Šmidrkal *et al.*, 2016). There are many potential sources of chlorine such as coagulants used to treat water

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(Chung, 2018), in which water is used for the generation of strip steam during deodorization. There are inorganic chlorides present naturally in palm fruit extract such as calcium chloride, magnesium chloride, and iron [II] chloride (Nagy *et al.*, 2011). The 3-MCPD formed in the refinery, especially during deodorization where high temperature (180 - 260°C) was introduced as both 3-MCPD and GE formed during heat-induced reactions. From 105 crude palm oils investigated, 80% of them contained no 3-MCPD esters but in refine, bleach, and deodorized (RBD) palm oil, only 1% of the samples contained 3-MCPD esters below the detection limit (Razak *et al.*, 2012).

There were debates on the free fatty acid (FFA) role in the formation of 3-MCPD esters, whereby some researchers claimed that during deodorization, the fatty acid and sodium chloride (could be any ionic bound chloride e.g. NaCl, KCl, TBAC) equilibrium is established and after that FFA salts and hydrogen chloride are formed (Šmidrkal *et al.*, 2016). Also, the 3-MCPD formation favours under acidic condition (Ramli *et al.*, 2011; Šmidrkal *et al.*, 2016). The MCPD fatty acid esters formation when the TAG, DAG, and MAG formed acyloxonium ion followed by nucleophilic attack of chloride ions (Rahn *et al.*, 2011; Šmidrkal *et al.*, 2016) as shown in Figure 1.

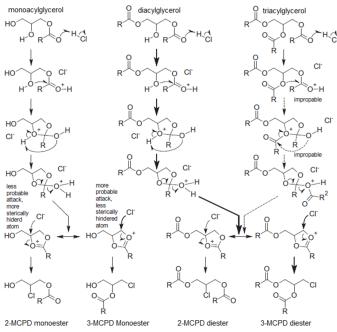
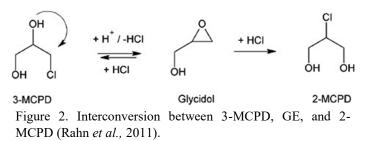


Figure 1. MCPD fatty acid esters formation mechanism (Šmidrkal *et al.*, 2016).

Apart from the formation of intermediate such as cyclic acyloxonium ion (Figure 1) or epoxside, the pathways of 3-MCPD ester formation involves direct nucleophilic attack to the carbon either carrying ester or hydroxyl group (Rahn *et al.*, 2011). The bidirectional

transformation also could happen between glycidol and 3 -MCPD or between their esterified forms in the presence of chloride ion as in Figure 2.



Partial acylglycerol such as DAG and MAG are the precursors of GE in refined oil (Cheng et al., 2017). The DAG and MAG can be formed from TAG through lipase hydrolysis or pyrolysis at high temperature (Shimizu, Vosmann and Matthäus, 2012). Unlike the MCPD esters, GE formed without the chlorine present; GE formation derived from intramolecular rearrangement through charge migration and different from each other depending on either the nature of the intermediate or the leaving group (Rahn et al., 2011; Destaillats et al., 2012b; Cheng et al., 2017). Cyclic acyloxonium ion, a well-known active intermediate in organic chemistry, may also represent in the formation of GE (Weißhaar et al., 2010). At the temperature range 180 - 230°C, only small content of GE detected but above 230°C, there is a steep increment of GE content was found (Hrnčiřík et al., 2011).

MCPD esters and GE formation are results of several competitive reactions which occur simultaneously. Even there is a competition between the same group forming a number of chloropropanol (CP) types, which explains why neither partial acylglycerol nor chlorinated compound could firmly correlate with the level of MCPD ester formed upon thermal heating (Ermacora *et al.*, 2014).

3. Mitigation strategies to minimize the 3-MCPD and GE formation

After understanding the precursors of 3-MCPD and GE formation, some researchers focused on how to reduce or eliminate the precursors such as removal of chlorin, partial acylglycerol, and free fatty acids (FFA). At the same time, others focused on changing the operation procedures, especially in the refining process, by introducing new steps of removing the formed MCPD esters and GE.

3.1 Field mitigation strategies

Several researchers related the inorganic chlorides in soils with the fertilizers used to support plant growth such as ammonium chloride and potassium chloride. Other than that, chloride possibly comes from the water irrigated to the farms. Both fertilizer and water will be absorbed by the plant for growth. The variation of the chloride-containing compound in the palm fruit depends on differences in the soil types, growth conditions, and genotype of the plants. So, the mitigation of MCPD esters needs as early intervention during cultivation by minimizing the use of substances such as fertilizers, pesticides, and irrigation water that have excessive amounts of chlorine-containing (Matthäus *et al.*, 2011).

The DAG, MAG, and FFA inside the fruit will increase when the TAG inside the mesocarp is hydrolyzed by lipase enzyme which also inside the fruit mesocarp. When the fruit ripes, the exocarp becomes soft and therefore makes it easy to be attacked by the lipase enzyme. Other than ripening factors, the lipase activity also accelerated by the damage of mesocarp tissue as a result of improper handling (Sambanthamurthi *et al.*, 2000). To minimize the FFA content, the fresh fruit bunch must be harvested before overripe with proper handling to prevent the bruise. The loose and damaged fruit should be collected for non-food purposes as it has low-quality oil. After harvesting, the fresh fruit bunches should be transported immediately to the factory for sterilization to inactivate the lipase enzyme.

3.2 Milling mitigation strategies

Sterilization will aid to stop the lipase activity as the enzyme inactive at high temperature which at least around 70°C for 40 mins within palm fruit (Shehu *et al.*, 2019), but conventional sterilization is performed at 140° C between 75 and 90 mins. According to Cheng *et al.* (2017), the sterilization temperature is better to be kept at or below 120°C (Cheng *et al.*, 2017). This is due to MCPD ester can be formed at a temperature of 120°C and higher (Zhao *et al.*, 2016).

The extraction of oil palm in the mill is usually done by mechanical means but Matthäus and Pudel (2014) suggested the use of a biphasic polar solvent (e.g. hexane -water) that can reduce the 3-MCPD diesters content. However, the adoption of this method will face toxicity of extracted oil and safety issues (i.e. high flammability of hexane), thus contrary to public acceptance.

Other than that, several researchers tested the forming of 3-MCPD from chlorine-containing CPO; it was washed with water (also with solvent), and then treated at high temperature (i.e. 230 - 240°C) to observe the formation of 3-MCPD (Matthäus *et al.*, 2011; Craft *et al.*, 2012). According to Matthäus *et al.* (2011), they found that after washing the CPO with water-ethanol of 25:75, showing that 3-MCPD esters, reduce to about 30% (compared to CPO without washing) after the heat

treatment, while the amount of GE was nearly unchanged, as shown in Figure 3. However, some drawbacks of washing the CPO using this method as it is costly, facing oil loss and emulsification properties own by phospholipids or other components lead to difficult separation.

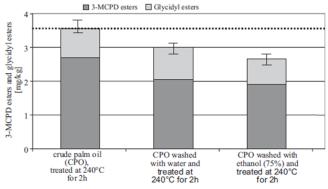


Figure 3. Effect of washing on the capability to form 3-MCPD esters and GE from CPO after the heat treatment (Matthäus *et al.*, 2011)

3.3 Refining mitigation strategies

The purpose of refining is to prepare the oil is edible; unacceptable components such as oxidation products, FFA, phospholipids, metals, trace of pesticide, and other impurities are removed (Ramli *et al.*, 2011). In general, the CPO can be refined through chemical refining process which involves four steps (i.e. degumming, neutralization, bleaching, deodorization) or via physical refining which consists of only three steps (i.e. degumming, bleaching, and deodorization). For the time being, the palm oil industry prefers physical refining because it gives advantage such as less environmental impact.

3.3.1 Before deodorization

Some of the researchers suggested improving the refining process in reducing the 3-MCPD and GE formation. For instance, the use of a low concentration of acid during degumming and the use of a natural type of bleaching earth instead of acidic bleaching earth (Vispute et al., 2018). Ramli et al. (2011) found that the formation of 3-MCPD ester at the highest level (up to 3.89 ppm) when using high dosage of phosphoric acid 0.1% (from tested range 0.012-0.1%) with a combination of acid-activated clay. The 3-MCPD esters formation were the lowest (0.25 ppm) when water degumming and natural bleaching clay was used. However, there is a drawback of using water degumming as it affects the oil quality due to insufficient removal of iron and phosphorus compounds. This work is suitable for using high-quality CPO. If the quality oil used is not good, it will have to be combined with another step, especially to remove the metallic impurities.

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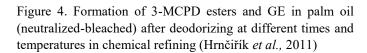
Some researchers suggested the neutralization step by adding calcium carbonate (Vispute *et al.*, 2018), calcium oxide (Ramli *et al.*, 2011), alkali potassium, or sodium bicarbonate (Šmidrkal *et al.*, 2016) to the degummed oil or during the bleaching process. On the other hand, Schurz (2010) proposed to use a high dosage of activated clay as it can absorb some of the 3-MCPD ester precursors from the oil. Besides, natural clay is recommended for the bleaching process, as mentioned before. However, natural bleaching earth may bring a negative effect on the colour of the oil. Chemical refining is seemed to be more attractive in the effort to reduce the 3-MCPD and GE as neutralization step offers pre-removal of FFA (Vispute *et al.*, 2018), thus improves the pH of the oil before deodorization.

The idea of adding additives in the bleached oil can prevent 3-MCPD formation; additives compete with precursors (e.g. chlorine-donating agent) in the reaction before stripping off during deodorization. Craft *et al.* (2012) added alcohol (e.g. ethanol, 2-propanol glycerol) to bleached palm oil, producing volatile chlorinated compounds that can be evaporated during deodorization, thus reducing the 3-MCPD esters.

Bertoli *et al.* (2011) described the effect of contacting carboxymethyl cellulose or cation exchange resin with oil resulting in a reduction of 3-MCPD content (comprises an amount of 1000 μ g or less per kg of oil) after contacting time of 1 hr at 60°C in a double jacketed glass reactor. The CPO was bleached using at least three absorbents such as 0.5% trisyl silica, 2% bleaching earth, and a suitable amount of ion exchange resin or carboxymethyl cellulose depending on the ratio of oil to resin. After the resin or carboxymethyl cellulose was removed, the bleached palm oil was deodorized using steam or nitrogen gas.

A patent provided by Zieverink et al. (2011)

□ 3-MCPD-E ■ Glycidyl-E



180

5h

230

1h

Temp [°C], time [h]

230

3h

230

5h

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5.0

4.5

4.0

3.5

3.0

2.5

2.0

1.5

1.0

0.5

0.0

180

1h

180

3h

Conc. [mg/kg]

discusses the fractionation of oil prior to deodorization whereby the solid high melting fraction (stearin) is separated from a liquid low melting fraction (olein). The deodorized palm oil fraction having 3-MCPD ester content of less than 1 ppm. However, there is no clear explanation of why fractionation can reduce MCPD esters.

3.3.2 Deodorization

According to Hrnčiřík et al. (2011), the deodorization conditions such as temperature (i.e. 180°C - 230°C) and residence time (i.e. 1 - 5 hrs), affected the level of GE but not to 3-MCPD esters within neutralizedbleached and bleached palm oil as shown in Figure 4 and Figure 5, respectively. Regardless of the deodorization conditions, the concentration of the 3-MCPD ester was more or less similar (3.5 - 4.7 mg/kg). On the contrary, GE concentration increased up to 2.2 mg/kg when the deodorization temperature at 230°C as shown in Figure 5. The results also show that lower deodorization temperature, 180°C, lower GE level was observed. However, it was not sufficient to remove FFA from the oil in which only 20% removal even deodorized for 5 hrs.

Dual deodorization had been discussed by Matthäus *et al.* (2014), to reduce the 3-MCPD esters and GE. The process is a combination of applying a longer deodorization time at lower temperature (i.e. 120 mins, 200°C), then followed by a shorter deodorization time at higher temperature (i.e. 5 mins, 250°C) or vice versa as stated in Figure 6. GE can be distilled or degraded at a higher temperature of 250 - 290°C or at a lower temperature of 200°C with longer deodorization time (e.g. 2 hrs), which is discussed by Craft *et al.* (2012).

Pudel *et al.* (2016) proposed a short path distillation instead of conventional deodorization. They reported that

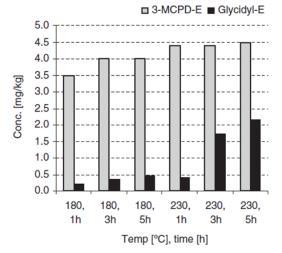


Figure 5. Formation of 3-MCPD esters and GE in palm oil (bleached) after deodorizing at different times and temperatures in physical refining (Hrnčiřík *et al.*, 2011).

different variables for the such as condenser temperatures, evaporator temperatures, stirrer speeds, and pump frequencies, there was no 3-MCPD ester was detected in any of the samples, while the level for GE at the limit of detection, 0.7 mg/kg. The optimum conditions in their works were condenser temperature at 60°C, evaporator temperature at 170°C, stirrer speed at 100 r/min, and pump frequency at 20 Hz. Matthäus et al. (2014) explained that short path distillation is one of the thermal separation techniques that taking advantage of high vacuum pressure (0.001-1 mbar) to lower the boiling point of substance, thus will facilitate the evaporation of undesired components.

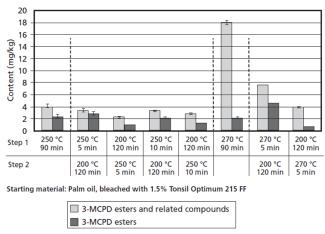


Figure 6. Effect of conventional and dual deodorization on 3-MCPD esters and related compounds formation (Matthäus *et al.*, 2014).

The presence of GE in RBD oil is determined by the rate of formation of GE and the rate of stripping of GE into the distilled phase. So, by knowing this, the refinery conditions can be modified by optimizing the parameters used (e.g. stripping steam rate, temperature, and pressure) in the steam distillation process during deodorization (Özdikicierler *et al.*, 2015). The authors said that there is significant interaction between stripping steam rate and temperature to the GE formation in their experiment.

Some researchers proposed the different types of stripping agents such as inert gas (Bertoli and Cauville, 2011) or acid solution (Matthäus *et al.*, 2011). When using an inert gas (i.e. nitrogen) as a stripping agent, they found that it will limit the formation of bound MCPD in oil as in Table 1. Bertoli *et al.* (2011) suggested the temperature of deodorization is between 140°C and 180°C, and under vacuum less than 50 mbar (preferentially less than 10 mbar). In addition, Siew *et al.* (2012) presumed that the inert gas prevents direct contact of chloro-compound with the oil. According to Matthäus *et al.* (2011), they replaced water with formic acid to generate strip steam and reduction almost 35% of GE in the RBD oil using a high concentration of formic acid

Stripping medium	3-MCPD µg/kg			
1 st steam trial	1340			
2 nd steam trial	1230			
1 st nitrogen trial	780			
2 nd nitrogen trial	710			

3.4 Post refining strategies

3.4.1 3-MCPD esters and GE removal from RBD oil

The 3-MCPD and GE can be removed from RBD oil by several methods such as physical adsorption, chemical degradation, and enzymatic reaction. Physical adsorption is mean to remove the 3-MCPD and GE by adsorbents such as activated carbon, magnesium silicate, zeolite, and activated bleaching earth without damaging their molecule structure by taking advantage of the fact that 3-MCPD esters and GE have different polarity from the oil. Strijowski et al. (2011) described that calcinated zeolite and synthetic magnesium silicate are possible absorbents in reducing polar materials such as DAG, MAG as well as the 3-MCPD esters and GE, with different transposition rates from both adsorption materials. As they found out that calcinated zeolite and synthetic magnesium silicate reached a maximum total reduction effected at different temperatures and times.

Shimizu *et al.* (2012) had used the acid-activated bleach earth (ABE) on RBD oil. They found out that rather than adsorption, there is a transformation of GE into glycerol mono-palmitate, glycerol palmitate oleate, and glycerol dipalmitate. This happens because of the GE that absorbed by ABE undergoes a molecular ringopening reaction. The last method to be discussed under this topic is the reduction of 3-MCPD and GE using the enzymatic treatment. In the aqueous system, the 3-MCPD converted to glycerol using enzyme consists of halohydrin dehalogenase from *Arthrobacter* sp. AD2 and an epoxide hydrolase from *Agrobacterium radiobacter* AD1 (Bornscheuer and Hesseler, 2010). According to them, *Candida antartica* lipase A also can convert 3-MCPD-esters to harmless glycerol in a biphasic system.

3.4.2 Storage

Matthäus *et al.* (2016) reported that the concentration of GE decrease when the oil stored at low temperature (5 - 15° C), as the crystallization process delivers enough energy to cleave the epoxy bonding of the GE. The degradation rate of GE per month at different temperatures is shown in Table 2. So, it is important in the production and in the distribution line to keep the oil in low-temperature storage or during

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Table	2.	Degradation	of	GE	per	month	at	different
temperatures (Matthäus et al. 2016).								

Temperature (°C)	Decrease glycidyl ester/month (mg/kg)
20	0.0
15	0.2
10	0.3
5	0.4
-20	0.1

4. Conclusion

The formation of 3-MCPD esters and GE greatly affected by the precursors such as chloride, DAG, MAG, and parameters such as temperature and pH. Build up some of the precursors should be avoided and eliminated. The mitigation step should be implemented for the whole chain of oil palm productions. Some of the mitigation procedures are expensive, thus become a turning point for many refiners to implement those approaches. The best practice scenario that can be adopted base on this review consists of several steps. The first step is to remove the precursors during the milling stage before extraction of the oil (e.g. use free-chlorine water, washing fruit from any pesticide or fertilizer residue, etc.), followed by the second step of selecting appropriate method and conditions during degumming. The final stage is to improve deodorization at optimal temperature and stripping rate; to minimize unwanted reactions and maximize the removal of 3-MCPD and GE.

Conflict of interest

The authors declare no conflict of interest.

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