TOTAL CHLORIDE CONTENT ANALYSIS IN CRUDE PALM OIL AND SECONDARY OILS

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The 3-monochloro-1,2-propanediol esters (3-MCPDE) are chemical food contaminants that are carcinogenic. The 3-MCPDE is formed due to high deodorisation temperature at the refining stage of crude palm oil (CPO). The primary precursor for 3-MCPDE formation is chloride which is present in the plantation and mill where it gets into the palm oil. Sources of chloride are water, fertilisers, pesticides, and soil (*Arris et al.*, 2020). Many studies show dependency between 3-MCPDE and total chloride content (TCC) in palm oil. A validated method for determining total chloride content (TCC) is critical for mitigating the presence of 3-MCPDE in refined palm oil products. Thus, MPOB has modified the official method (ASTM D4929) analytical procedure and validated it to determine TCC in CPO and secondary oils derived from empty fruit bunch oil, palm press fibre oil, palm oil mill effluent, and sludge palm oil using a Total Chloride Analyzer (TCA). TCA consists of an automatic combination of oxidative combustion and coulometric titration that is simple to use and can produce accurate data.

THE TECHNOLOGY

Principle

The principle of TCA analysis is based on combustion followed by coulometric titration. The sample was either injected or placed into a combustion boat where the temperature was set at 1000°C. Argon and oxygen were passed through the boat and any chlorine containing components were completely combusted. The resulting combustion products were swept into the titration cell. The electrolyte solution containing silver ions was immediately combined with any chloride ions. The amount of chloride was calculated from the quantity of electricity required for titration. (Mitsubishi Chemical Analytech, 2014)

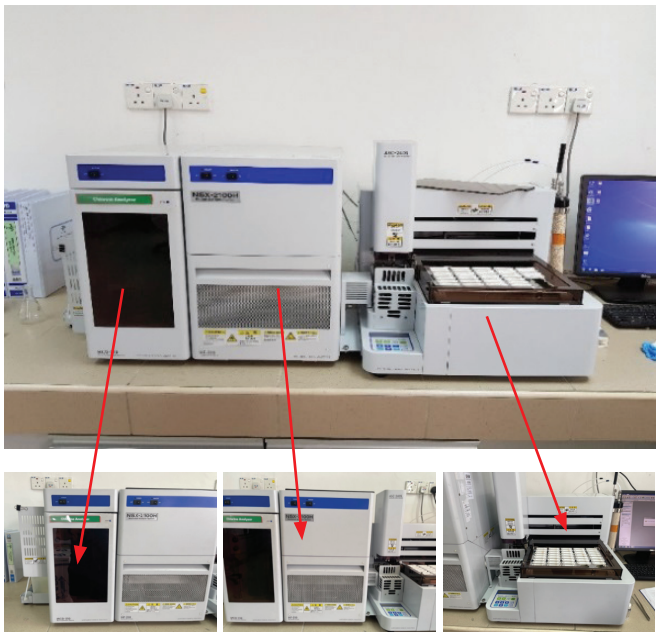

Figure 1. TCA instruments for the determination of TCC.

Method Validation

Method development has been carried out to determine TCC in CPO and secondary oils. The combustion and titration conditions for the TCA are shown in *Table 1*. The results indicate good linearity from 0.5 µg mL^{-1} and 20 µg mL^{-1}, while the correlation coefficient (R^2) was more than 0.999 (*Figure 2*). The average recoveries for CPO, empty fruit bunch oil, palm press fibre oil, palm oil mill effluent, and sludge palm oil at three spike levels were between 85% and 105% with the relative standard deviations (RSD) of less than 10%. The limit of detection (LOD) and limit of quantification (LOQ) were estimated at 0.05 µg mL^{-1} and 0.18 µg mL^{-1}, respectively (*Table 2*).

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TABLE 1. OPERATING CONDITIONS OF TCA FOR COMBUSTION PROCEDURE

Parameter	Condition
Reactant gas flow, Oxygen	200 mL min^{-1}
Carrier gas flow, Argon	200 mL min^{-1}
Furnace temperature inlet	1000°C
Furnace temperature outlet	1100°C
Sample amount	40 mg to 60 mg
Detection mode	Coulometric titration

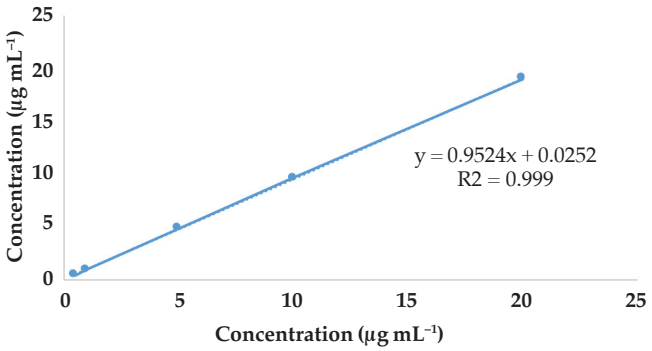

Figure 2. Calibration curve for the determination of TCC analysis

TABLE 2. METHOD PERFORMANCE OF TCC ANALYSIS

Parameter		Data
Calibration curve		y = 0.9524x + 0.0252
R2		> 0.999
LOD		0.05 µg mL^{-1}
LOQ		0.18 µg mL^{-1}
Recovery study	0.5 µg mL^{-1}	87% to 102%
	1.0 µg mL^{-1}	85% to 103%
	5.0 µg mL^{-1}	90% to 105%

BENEFIT

Availability of a simple and reliable method for detection and quantification of TCC in CPO and secondary oils.

SERVICE OFFERED

- Analytical test service for TCC in CPO and secondary oils.
- Training session for the analysis of TCC.

INDUCTIVE COST

The test service cost RM300 per sample, whereas the training session cost RM18 000 per person. The cost is subject to change without prior notice.

REFERENCES

Arris, F A; Thai, V T S; Manan, W. N and Sajab, M S (2020). A revisit to the formation and mitigation of 3-chloropropane-1,2-diol in palm oil production. *Foods, 9 (12)*: 1769.

Mitsubishi Chemical Analytech (2014). Operational manual (horizontal furnace) for coulometer Model MCD-210.

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