DETERMINATION OF THE EUTECTIC MIXTURE OF DIPHENYL OXIDE AND BIPHENYL IN VEGETABLE OILS AND OLEOCHEMICALS BY HPLC-DOWTHERM A™

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MAY 2002

179

MPOB TT No. 167

MPOB INFORMATION SERIES ISSN 1511-7871

A survey (Tang *et al*., 1996) to gather information on the types of heating media used in the Malaysian palm oil refineries carried out by the Malaysian Palm Oil Board (MPOB) in 1995 revealed that a majority of the older palm oil refineries still use thermal heating fluids as heat-transfer media, but none of the fluids used are certified as food-grade. Even though the authority has recommended the replacement of thermal heating fluids by high-pressure steam as an alternative heat-transfer media, such move was slow as the cost involved is rather high.

The survey also detailed that out of 41 refineries operating in 1995, 20 plants were still using thermal heating fluids as heating media in the deodorizers, and only 12 plants used high-pressure steam. The remaining nine plants used a combination of thermal heating fluid and high- and low-pressure steam in either batch or continuous processes.

In view of a sizeable usage, the safety of the thermal heating fluids as heat exchange media in edible oil processing has always been an issue. Although there were attempts to forbid the use of thermal heating fluids in edible oil processing plant (Rossell, 1993), the Codex Committee on Fats and Oils had resolved to allow the use of this heating media other than high-pressure steam on the basis of safety and risk evaluation and inspection procedures (Codex, 1999). As a result, the local industry has requested MPOB to look into such matters to develop relevant analytical methods for the detection of thermal heating fluids in various palm oil products which can be used for quality control purposes.

ANALYTICAL STATUS

Reports (Rossell, 1993; Tang *et al*., 1995) showed that there are several brands of petroleum-based thermal heating fluids available in the market nowadays. One of the commonly used thermal heating fluid is the highly stable eutectic mixture of diphenyl oxide and biphenyl, which is traded as Dowtherm A™ and Therminol VP-1™.

The crude method for detecting contamination is based on its characteristic smell or a drop in the pressure in the system. However, this method is qualitative and not reliable.

On the other hand, gas chromatography (GC) is quantitative and has offered a more sensitive means for detecting the eutectic mixture after some pre-treatment steps. Takagi (1969) has reported a GC method for the determination of the eutectic mixture in fatty oils after separation of hydrocarbon fraction from the unsaponifiable materials by thin-layer chromatography. Imai *et al*. (1974), described the method that incorporate a pre-distillation to recover the eutectic mixture before GC analysis. Min and Wen (1982) took a step further by installing a U-shaped precolumn before the GC column to trap the oil sample, thus avoiding the sample preparation. The standard American Oil Chemists' Society (AOCS) method Cd-25-96 for the detection of the eutectic mixture by GC also requires multiple solvent extractions. Although the detection limits of these GC methods are at the range of 0.2-5.0 ppm levels, the procedures are time-consuming and tedious.

METHOD DEVELOPED BY MPOB

A new high-performance liquid chromatographic (HPLC) method with fluorescence detection has been developed for the determination of the eutectic mixture in vegetable oils and oleochemicals (Moh and Tang, 1999; Moh *et al*., 2000). The HPLC method is superior to those GC methods previously reported in that it requires no pre-treatment - a 1 g sample is weighed into a 10 ml volumetric and diluted with tetrahydrofuran. A 20 µl of the aliquot is then analysed by HPLC directly.

System Requirement

1. HPLC system - with minimum requirements of a high-pressure pump, sample injection device, fluorescence detector (247 nm excitation and 310 nm emission), and chart recorder.

2. HPLC analytical column - 250 mm x 4.6 mm i.d., with 5 µm reversed-phase LiChrospher C18 (GL Sciences Inc., Tokyo, Japan) or equivalent.

3. Column temperature - maintained at 30°C with a column oven.

4. Injection syringe - capable of accurately injecting 20 µl.

METHOD STATUS

The HPLC method has been accepted by the AOCS as the Recommended Practice Cd 25a-00.

SERVICES

1. Consultation - assist in setting up HPLC system for the analysis and provide training.

2. Analytical service - welcome samples for cross checking.

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