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Research Article

Simultaneous profiling of methanol and ethanol in local and foreign alcoholic beverages by GC-FID

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Abstract
Intoxication with methanol from alcoholic beverages is a global issue. Methanol can cause severe health complications and death. For these reasons, numerous methods for measuring methanol in alcoholic beverages have developed. This study aimed to develop an accurate, sensitive, and simple method for determining methanol and ethanol in local and foreign alcoholic beverages using GC-FID. The method was developed by optimising GC parameters including an initial oven temperature of 40°C held for 2 minutes, ramping at 25 °C/min until 100°C, a carrier gas flow rate of 4.5 mL/min, a 2:1 split ratio, and an injection volume of 10 µL. The optimised method met UNODC guidelines for specificity, linearity, detection and quantification limits, precision, and accuracy. Both the methanol (0.001%–0.625%) and ethanol (5%–25%) calibration curves had correlation coefficients (r^2) of more than 0.99. LOQs for methanol and ethanol were 1.72 and 1.76 mg/L, respectively, with good precision and recoveries. The run time was 5.40 minutes to complete. 19 different alcoholic beverages were analysed by injecting them directly into the GC-FID after adding acetonitrile as an internal standard. This study revealed that 36.8% of samples had methanol concentrations between 0%–0.0198%, while ethanol was detected in every sample. The proposed method is sensitive, simple, and requires no pre-treatment, making it suitable for forensic toxicological analysis. Importantly, methanol was reliably detected without interference from higher concentrations of ethanol. This research demonstrated that the DB ALC 1 column capillary in GC-FID with optimised parameters is very selective and sensitive in measuring methanol and ethanol simultaneously in alcoholic beverages.

Keywords: forensic toxicology, ethanol, methanol, alcoholic beverages, GC-FID

Introduction

Malaysia is a Muslim country that adheres to Islamic law. Most Malay eateries don't serve alcohol. However, non-Muslims can still buy alcohol at Western-style hotels, pubs, and restaurants. This contributes to Malaysia's high alcohol consumption especially among adolescents [1]. According to nationwide study on alcohol consumption [2], most Malaysians prefer beer. Malaysia's excise duty on beer is the second highest in the world, after Norway. If the excise duty is raised, the product price will rise, encouraging the growth of black market. Hard-core drinkers who cannot afford beer will drink illicit beverages because they are cheaper. The main concern with illicit alcoholic beverages is whether they are homemade or produced illegally. These products may produce new toxic alcohols, repackage popular brands with cheaper alternatives, or add unrecorded compounds. One of the unrecorded compounds is methanol, a sweeter but more harmful alcohol. Methanol can be produced in alcoholic beverages naturally, through pectin degradation in fermented fruits [3], or artificially, through adulteration by illegal addition of the pure compound

[4]. Therefore, most fermented alcoholic beverages contain methanol in addition to ethanol.

However, methanol in alcoholic beverages has maximum residual limits (MRL), and exceeding these limits can result in toxicity. The European Union (EU) sets the legal limit for methanol in alcoholic drinks at 0.4% (v/v) [5]. As of now, Malaysia's Food Regulations 1985 and the related amendments do not specify a maximum residue limit (MRL) for methanol in alcoholic beverages and relies on the general food safety provisions and the post-market enforcement. Consequently, methanol poisoning incidents from illicit alcohol remain a recurrent public health issue in Malaysia. The ingestion of 10 mL methanol can cause blindness while 30 mL can be fatal [6]. For instance, Malaysia experienced methanol poisoning outbreaks in early September 2018. According to Muhammad Adil *et al.* (2019) [7], six people died from severe metabolic acidosis due to counterfeit liquor with most drinkers had stomach problems and headaches. Other nations experienced the same problems as previously reported, including 20 deaths in Costa Rica and 59 in India in 2019 [8,9]. In 2018, six Egyptians had metabolic acidosis and visual impairment [10]. In the same year, 84 of 795 Iranians died with 314 patients showed gastro-intestinal symptoms, 57 had loss of consciousness, and five became legally blind [11].

Methanol poisoning from alcoholic beverages has been a common issue for years. Hence, various techniques have been developed for determining alcohol concentration levels. For a long time, hydrometers [12], pycnometers [13], and electronic density metres [14,15] have been officially recognised as methods for measuring alcohol proof and tax declaration in product labelling. These methods are inexpensive and do not require standards or chemicals. However, they provide inconsistent results and are unsuitable for samples containing trace amounts of alcohols. In order to circumvent these obstacles, the alcohol content of beverages is measured using sensitive, rapid, and simple techniques, such as Fourier transform infrared spectrometry (FTIR) [16], high-performance liquid chromatography (HPLC) [17], Raman spectroscopy, nuclear magnetic resonance spectroscopy (NMR), non-invasive near-infrared (NIR) [18-20], UV-visible spectrophotometry [21], and direct analysis in real-time mass spectrometry (DART-MS) [22]. However, these methods typically require time-consuming sample preparation, prone to interference peaks, and demand trained analysts to operate.

Gas chromatography (GC) is the gold standard and most widely used technique for alcohol analysis. This instrument can separate and measure complex matrices like methanol and ethanol [23]. **Table 1** illustrates several GC techniques that detect adulterated alcohol. Most of these techniques suffer a lack of sensitivity to detect trace amounts of methanol in highly concentrated alcoholic beverages. In light of this, an attempt was made in the current study to develop a method using a DB ALC 1 capillary column in conjunction with a GC technique to address some of the aforementioned limitations. This method was expected to be simple, accurate, sensitive, and highly reproducible, and it required no pre-treatment. The aim of this study is to simultaneously determine methanol and ethanol in local and foreign alcoholic beverages in Malaysia using GC-FID.

Materials and Methods

Beverage samples

Nineteen (19) samples of local and foreign alcoholic beverages were purchased in Pahang, Melaka, and Selangor. Some samples seized by the Pahang Customs Department were also collected for analysis. All samples were kept at 4°C in a refrigerator prior to analysis.

Reagents and chemicals

Absolute ethanol standard (99.6%) was sourced from Merck (Germany), and an absolute methanol standard (99.6%) was obtained from Fisher. Acetonitrile (99.9%) which was used as an internal standard (IS), together with n-butanol and n-propanol were obtained from Merck (Germany). The deionised water used in standard preparations, had a resistivity of 18.2 mΩ/cm, supplied by Elga Purelab Option DV25 (USA).

Standard preparations

For instrument optimisation, mixed standard solutions (0.1% w/v of methanol, ethanol, and acetonitrile) were prepared by transferring 0.1 g of absolute methanol, ethanol, and acetonitrile into a 100 mL volumetric flask and filling it to the target mark with deionised water. For the validation study, five concentration levels of working standard solutions were prepared for the calibration curves by mixing a series of concentrations (0.001%–0.625% methanol and 5%–25% ethanol) into a volumetric flask and topping up to the target mark with deionised water.

Apparatus and instrumentation

Flasks, pipettes, test tubes, and 2 mL GC vials of grade A were used. A Whatman 0.45 µm PTFE syringe filter was used to filter samples. The following micropipettes were used: Eppendorf Research (20–200 µL), Finnpipette Thermo (100–1000 µL), Eppendorf Research (1–5 mL), and Eppendorf Research (1–10 mL). For sample preparation, a Sartorius CP224S analytical balance, a Barnstead Thermolyne vortex mixer, and a Rotofix 32A centrifuge were used.

Table 1. The performance of various gas chromatography techniques in alcohol analysis

Extraction/ Instrument	Column	Determined Compounds	Run Time (min)	LOD (mg/L)	Reference
GCFID	CP-WAX 57 CB	Methanol	21	10.8	[24]
GCFID	Restec MXT-1	Ethanol	6	800	[25]
GCFID	HP-FFAP	Ethanol, methanol, impurities	22	10	[26]
GCMS	HP-FFAP	Methanol and its derivatives	24.7	0.0008	[27]
HS-SPME/GCMS	HP-INNOWAX	Ethanol	14	30	[28]
GCFID	DB-WAX	Methanol, other alcohols	30	1.84	[29]
GCFID	RTX-WAX	Methanol, ethanol	22	2.87, 2.24	[4]
SPE/GCFID	DB-624	Methanol, ethanol	16	1	[3]
HS-GC-FID	Zebron BAC 1	Ethanol	2.4	6	[30]
GCFID	CARBO PACK B	Ethanol	16	670	[31]
GCFID with magnetic stirring-assisted aqueous extraction	HP-INNOWAX	Ethanol	25	0.3	[32]

In this work, an Agilent model (G 1540N) 6890 gas chromatography instrument, combined with a flame ionisation detector, was utilised. DB-ALC1 column (J&W 125-9134) was used to separate analytes. The detector and back inlet port temperatures were both set to 250°C at 3.34 psi. Helium as a carrier gas was set in constant-flow mode. The flow rates of hydrogen and air were set at 40.0 mL/min and 450.0 mL/min, respectively. Nitrogen was applied as the makeup gas in a set mode under a combined constant column, with a makeup flow of 45.0 mL/min. All samples were automatically injected in split mode using a 10 µL syringe. This analysis was performed using Agilent ChemStation software (G2080BA, revision code B.01.03).

GC-FID method development

The GC-FID method was developed to simultaneously detect methanol and ethanol. **Table 2** lists the optimisation parameters and their settings tested in this study. The use of acetonitrile, n-propanol, and n-butanol as IS in this method was also investigated.

GC-FID method validation

This optimised and developed method was validated according to UNODC recommendations. The method was validated for specificity, linearity, detection limit, quantification limit, precision, accuracy, and recovery under optimal conditions as well as system suitability. Integrated peak areas were used to measure analyte concentrations because they are stable and reproducible. To ensure accurate results, all samples and calibrations were replicated.

Specificity

Five real samples were used in this study (alcoholic beverage, energy drink, tea-based beverage, fruit-based beverages, and Coway drinking water). The presence of methanol and ethanol peaks in the real samples was confirmed by retention time comparison with a spiked reference sample. This study also examined potential interference in these samples.

Table 2. Optimisation of GC parameters

GC parameters	Setting programme
Optimisation of initial oven temperature and initial time	Range from 40 to 90℃ Range from 0 to 2 minutes
Optimisation of final oven temperature	Range from 100 to 200℃
Optimisation of heating rate	Range from 15 to 25℃/min
Optimisation of mode of inlet	Range from splitless to split ratio of 2:1 and 5:1
Optimisation of flow rate of carrier gas	Range from3 to 10 mL/min
Optimisation of injection volumes	Range from 0.2 to 2.0 µL

Detection and quantification limits

LOD and LOQ were calculated using signal-to-noise ratios (S/N) according to equations (1) and (2). LODs for methanol and ethanol were determined by analysing spike samples with gradual concentration reductions. LODs are three times the signal-to-noise ratio (S/N = 3) while LOQs are ten times.

$$\text{LOD} = 3 \times (\text{concentration} / \text{signal to noise ratio}) \quad \text{(Eq.1)}$$

$$\text{LOQ} = 10 \times (\text{concentration} / \text{signal to noise ratio}) \quad \text{(Eq. 2)}$$

Linearity

Two calibration curves were plotted in the ranges of 0.001% to 0.625% for methanol and 5% to 25% for ethanol, using linear regression of peak area ratios of analytes and internal standards against their concentrations (%). After constructing calibration curves, correlation coefficients (r^2) and intercept values were used to determine the linearity of methanol and ethanol.

Precision

This study measured precision under repeatability and reproducibility conditions. Repeatability was calculated using intra-day precision with 11 replicates of a single mixed standard concentration (n = 11). Reproducibility was measured by analysing low, medium, and high concentrations in triplicate for over three days (n = 27). As shown in equation (3), precision is measured by the correlation coefficient or relative standard deviation (RSD).

$$\text{Precision, s} = \sqrt{\frac{\sum(x_i - \bar{x})^2}{n-1}} \quad \text{(Eq. 3)}$$

Accuracy

The accuracy was determined by analysing triplicate samples spiked with known quantities at three different concentrations (low, medium, and high). The deviation between the mean result of spiked samples and that of expected reference (c) was calculated, as expressed by the equation (4).

$$\text{Accuracy, error (\%)} = \frac{c - \bar{x}_{measured}}{\bar{x}_{measured}} \quad \text{(Eq. 4)}$$

Recovery

The recovery study was carried out by making samples with known amounts of mixed standards at three concentration levels (low, medium, and high) in a matrix of samples (alcoholic and non-alcoholic beverages). Triplicates of each spiked or fortified sample were analysed after adding a known amount of internal standard (1%). At the same time, unfortified samples were also tested. The recovery was calculated by comparing the differences in peak area of the target analytes between fortified and unfortified samples to the peak area of the standard solution or fortification, expressed in percentage, as shown in equation (5).

$$\text{Recovery, Rec (\%)} = \frac{c_{fortified} - c_{unfortified}}{c_{fortification}} \quad \text{(Eq. 5)}$$

GC-FID method application

The established method was applied for the analysis of 12 local alcoholic beverages and seven foreign alcoholic beverages with varying levels of alcohol. All samples were collected from different brands and labelled as L1 to L12 for local brands and F1 to F7 for foreign brands.

Each beverage sample was filtered to remove solid impurities. 1 mL of filtered sample was dispensed into a GC vial, followed by 100 µL of IS. The vial was vortexed to mix the contents before being centrifuged

for 5 minutes at 2500 rpm to remove any remaining solid impurities. Each sample was prepared in duplicate. 1 µL of the sample solution was then injected directly into the column.

The auto-sampler was programmed to rinse each injection syringe with distilled water to prevent cross-contamination. The method was also checked by using quality control (0.1% methanol and ethanol) at the beginning and end of the sample sequence. Only sample results within QC limits are accepted. Peaks of methanol, ethanol, and IS were identified. The concentration (%) was calculated using GC software based on the calibration curve and obtained directly from the GC reading. If the difference between the first and second samples exceeded 5% RSD, the sample was remeasured.

Results and Discussion

Experimental GC conditions

The GC-FID was successfully developed under optimal conditions to simultaneously detect and quantify methanol and ethanol, as summarised in **Table 3**.

Initial oven temperature and initial hold time

The initial oven temperature and hold time were programmed to facilitate early elution of analyte peaks with good separation. To improve the peak separation, the initial oven temperature should be reduced rather than adding an initial hold time. At 90°C, no peaks were detected within the allotted time. At 40°C, the separation of analyte peaks was better than at 60°C, although the analysis time increased with longer hold time. However, higher oven temperatures shortened the analysis time [32]. Therefore, the optimal condition was chosen at 40°C with 2-minute hold, since it produced better separation within a reasonable timeframe.

Final oven temperature

The final oven temperature affected both analyte peak elution and analysis duration. The hold time was optimised until no more peaks were observed. While final temperature settings impacted analyses time, peak separation and retention times were not affected. Higher final temperature reduced run time analysis. As a result, 100°C was chosen as the optimal final temperature since it provided the shortest run time analysis while maintaining good separation.

Heating rate

The heating rate affected the resolution of analytes that elute in the middle of the chromatogram. The elution of analytes became faster as the ramping rate increased, resulting in a shorter analysis time. At 25 °C/min, the optimal configuration achieved the shortest run time, with high resolution that clearly separated methanol, ethanol, and IS.

Mode of inlet

The inlet mode affects method sensitivity based on analyte response. Split mode is typically used for small amounts, while splitless mode is applied for high concentrations. Both injection modes were investigated to determine trace amounts of methanol and highly concentrated ethanol. In Figure 1, as the mode was changed from splitless to split and the split ratio increased, the peak area of analytes decreased. However, across all three conditions, the peak area ratio of each analyte gave the same results when normalised by IS calibration mode. This indicates that the method sensitivity, as measured by the peak area ratios, was unaffected by inlet mode. There was no difference in analyte responses between splitless, split ratio 2:1 and 5:1 because $p>0.05$. However, split mode was chosen in order to extend the lifetime of the liner and column.

Table 3. Parameters of GC-FID method developed in this study

Parameter	Condition
Carrier gas	Helium
Mode of carrier gas	Constant flow
Flow rate of carrier gas	4.5 mL/min
Injection volume	1.0 µL
Injection mode	Split ratio 2:1
Inlet temperature	250°C
Oven temperature	40°C hold for 2 min and ramp up to 100°C at a rate of 25 °C/min and hold for 1 min
Detector temperature	250°C
Hydrogen flow	40 mL/min
Air flow	450 mL/min
Combined flow	45 mL/min
Total run time	5.40 min

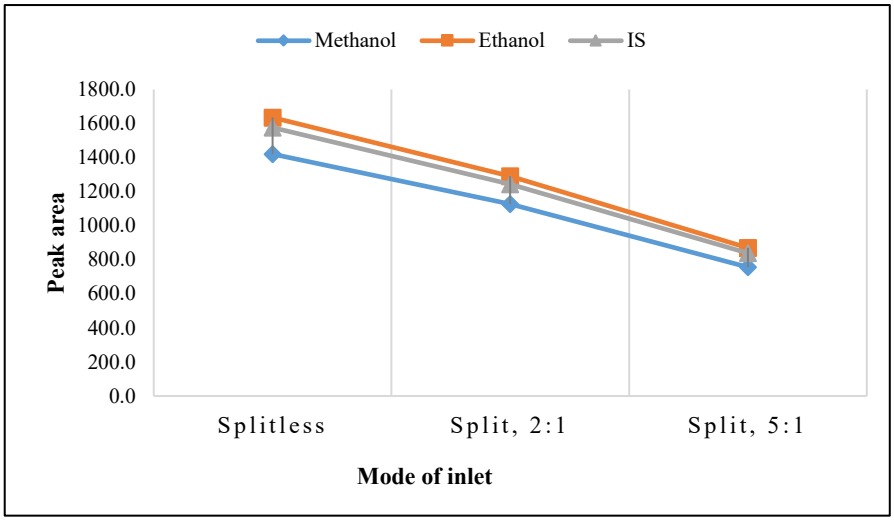

Figure 1. Effect of inlet selection mode on analytes' peak area

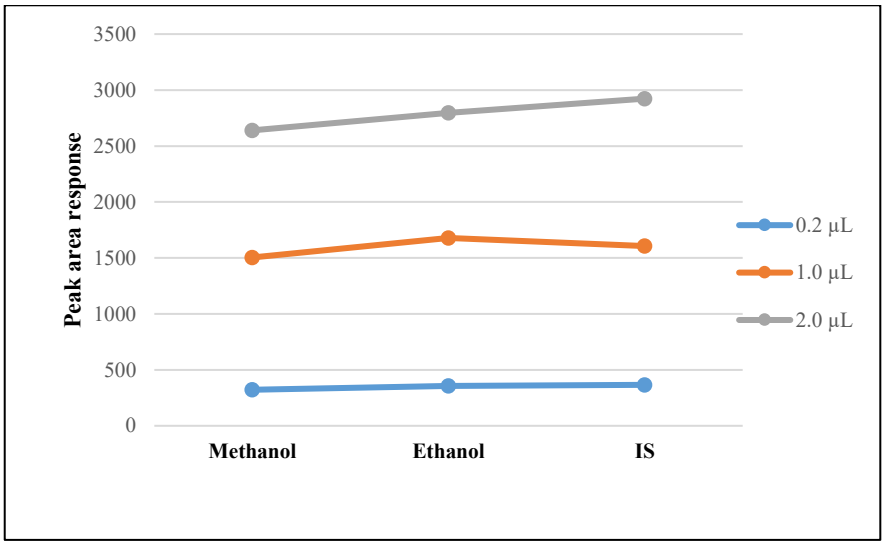

Figure 2. Effects of injection volume on peak area response of analytes

Flow rate of carrier gas

The carrier gas flow rate determines elution speed and column separation efficiency. Both flow rate and inlet mode were tested to improve column separation. No peaks were detected at 1 mL/min and a 2:1 split ratio. As flow rate increased, analyte peaks eluted faster, but the distance between peaks narrowed, resulting in poor separation. A flow rate of 4.5 mL/min with a split ratio of 2:1 was found to be optimal for good peak separation.

Injection volume

The syringe injection volume affects the sample volume, which in turn affects the method sensitivity through peak area response. **Figure 2** demonstrates that injection volume increased with the injection volume. However, a peak shoulder and tailing appeared when injecting 2.0 µL. An injection volume of 1.0 µL was chosen as the optimal setup, providing optimal sensitivity and reproducibility.

Selection of internal standard (IS)

The addition of internal standard (IS) improves assay precision and accuracy. No overlap was observed between IS and analyte peaks. Comparatively, acetonitrile's peak was closest to ethanol's. Acetonitrile was chosen as the IS because it is stable, absent in alcoholic beverages and has volatility

comparable to methanol and ethanol, ensuring elution within a suitable retention time window. It produces sharp, well-resolved peaks in GC analysis, improving peak integration accuracy. Furthermore, acetonitrile has been recommended as an IS for volatile analysis in alcoholic beverages because of its robustness and reproducibility [23]. In contrast, due to their potential presence in alcoholic beverages, n-butanol and n-propanol were unsuitable for alcohol analysis.

Performance of validation parameters

An optimised method with high accuracy and sensitivity was successfully validated. The validation performance of the developed method is summarised in **Table 4**.

Specificity

Specificity refers to a method's ability to accurately identify analytes in a sample matrix under optimal conditions [30]. The specificity was determined by measuring the retention times of methanol, ethanol, and IS in beverage samples. As shown in **Figure 3**, this method produced excellent chromatographic specificity for methanol, ethanol, and IS with retention times of 2.575, 3.193, and 3.893 min, respectively. The method was selective for the target compounds because their retention times did not overlap, and no interfering compounds were observed.

Detection and quantification limits

The limit of detection (LOD) is the lowest concentration of an analyte in a sample that can be detected but not necessarily quantified. On the other hand, the limit of quantitation (LOQ) is the lowest concentration that can be measured [31]. The sensitivity of the GC method was evaluated by determining the LOD and LOQ for methanol and ethanol, as presented in **Table 4**. Based on this outcome, the developed method demonstrated high sensitivity and capable of detecting methanol concentrations as low as 0.0002%.

Linearity

Linearity describes a method's ability to produce test results proportional to the analyte in a given range [33]. **Figure 4** depicts the calibration curves for the methanol and ethanol in this study. The correlation coefficients (r^2) obtained for methanol and ethanol were 0.9999 and 0.9995, respectively, indicating good linearity.

Table 4. Validation parameters performed in developed method

Parameter	Methanol	Ethanol
Linear range (%)	0.001 – 0.625	5 - 25
Correlation coefficient (r^2)	0.9999	0.9995
LOD (mg/L)	0.52	0.53
LOQ (mg/L)	1.72	1.76
Repeatability (% RSD)	1.27	1.37
Reproducibility (% RSD)	<1.69	<2.02
Accuracy (% error)	<1.78	<1.68
Recovery (%)	100.47 – 108.70	85.50 – 105.90

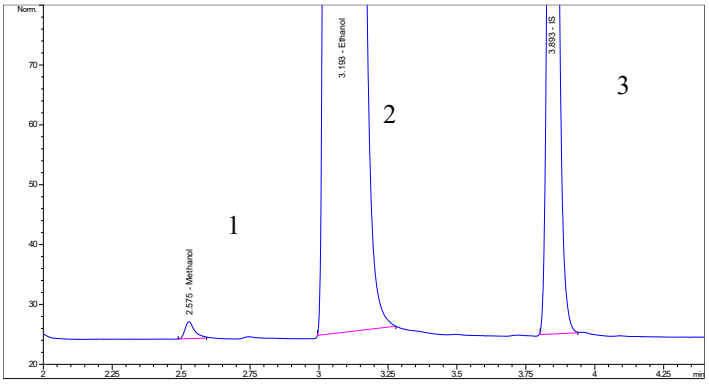

Figure 3. Representative chromatograms for specificity study in spiked sample. 1: Methanol; 2: Ethanol and 3: IS.

a) Methanol (0.001 – 0.625 %) b) Ethanol (5 – 25%)

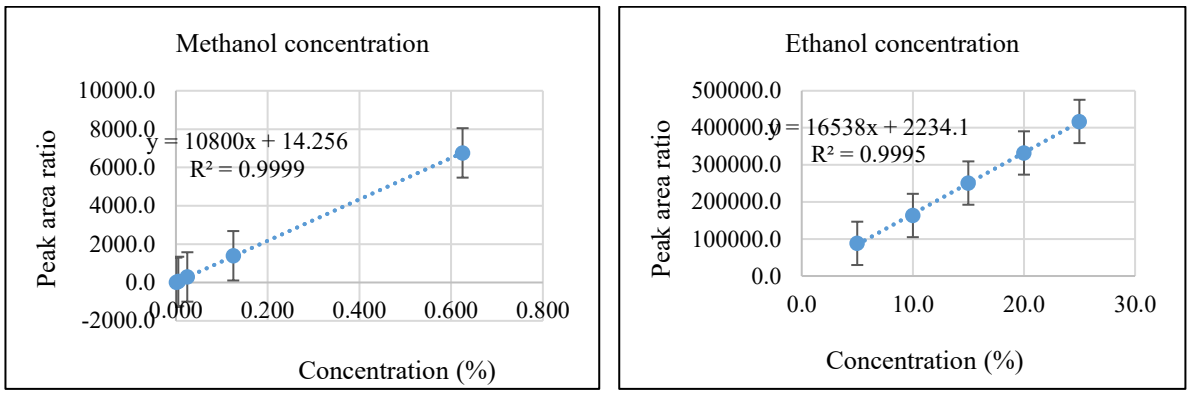

Figure 4. Calibration curves of analytes by using GC-FID using optimal conditions. (a) Methanol and (b) Ethanol

Precision

Precision refers to the degree of agreement among replicate measurements of the same analyte performed under the optimal method's conditions [34]. The repeatability and reproducibility of the method are summarised in **Table 4**. For both methanol and ethanol, low % RSD values indicated high precision, validating that the developed method consistently measured both analytes.

Accuracy

The accuracy is defined as the difference between the measured test result of target substances and the accepted reference value, typically expressed as a percentage [35]. Based on **Table 4**, the experimental results revealed a small percentage error, indicating that the measured value of analytes were close to the target values. This study demonstrated that the developed method accurately measured all target substances.

Recovery

Recovery studies involve adding a known amount of analyte to a sample and determining the proportion recovered [36]. In this study, three concentration levels of mixed standards were spiked into samples of alcoholic (Apple Fox Cider) and non-alcoholic (You C1000 Vitamin Lemon Drink) beverages, followed by analysis with the established method. Under optimal GC conditions, each sample was directly injected into GC, and the recovery was then calculated based on the peak area of each analyte. The % recovery values are presented in **Table 4**. All of these results complied with UNODC validation requirements, which state that recovery should be reproducible within ±15%, or between 85% and 115%.

Application of validated method

The mean results from two replicates of each sample of alcoholic beverages are presented in **Table 5**. Of the 19 analysed samples, 36.8% contained detectable levels of methanol, ranging from undetectable to 0.0198%. This study found that 71.4% of the tested positive methanol was found in wine rather than beer. All methanol concentrations detected were below the EU-mandated legal limit [5]. Ethanol concentrations were accurately calculated across the full range, from the lowest to the highest concentration (up 40% v/v), by direct injection and without dilution. The %RSD between replicates ranged from 0.01%–1.88% for ethanol and from 0.19%–2.29% for methanol, proving that the established method precisely analysed the samples.

Conclusion

The GC-FID developed method was optimised for sensitivity and time required for analysis. Initial oven temperature of 40°C held for 2 minutes, ramping at 25 °C/min until 100°C, a carrier gas flow rate of 4.5 mL/min, a 2:1 split ratio, and an injection volume of 10 µL were selected as optimal conditions. Methanol and ethanol calibration plots were linear with $r^2 >$ 0.99. The LODs for methanol and ethanol were 0.52 mg/L and 0.53 mg/L, respectively. The established method was applied to 19 samples of local and foreign alcoholic beverages and found that 36.8% of the samples contained methanol concentrations, ranging from undetectable to 0.0198%, while ethanol was present in all samples. A trace level of methanol was reliably detected without interference from higher ethanol levels. Samples can be analysed directly in 5.40 minutes without any pre-treatment.

Table 5. Summary of the mean concentrations of methanol and ethanol in the analysed alcoholic beverages

Sample Label	Ethanol		Methanol	
	Mean Concentration (%)	% RSD	Mean Concentration (%)	% RSD
L1	4.7198	0.49	-	-
L2	5.3713	0.50	-	-
L3	4.7377	0.50	0.0011	1.20
L4	4.4071	0.01	-	-
L5	31.0612	0.30	0.0013	1.36
L6	20.4960	1.54	0.0015	1.88
L7	39.3043	1.75	-	-
L8	29.1485	0.12	-	-
L9	30.9441	0.21	-	-
L10	12.4739	0.54	0.0008	0.19
L11	11.4561	0.34	0.0007	2.29
L12	12.4403	1.47	0.0009	0.55
F1	4.4095	0.09	-	-
F2	3.9828	0.51	-	-
F3	4.6752	0.38	-	-
F4	4.4976	0.68	-	-
F5	41.2561	0.37	0.0198	0.30
F6	43.9534	0.19	-	-
F7	13.1754	1.88	-	-

However, this study was limited by gas chromatography–related issues such as contamination, carry-over, and column bleed when analysing high-ethanol, multicomponent samples. In this study, only several local brands of alcoholic beverages were tested, and all methanol levels found were below the EU legal limit. Notably, the Food Act 1983 and Food Regulations 1985 do not specify acceptable methanol levels in alcoholic beverages in Malaysia. Therefore, as a future recommendation, this method should be applied to a larger number of local alcoholic beverages in Malaysia to support clinical investigations of methanol-related deaths. The findings may also contribute to establishing a tolerable limit for methanol in Malaysia's food regulations. Further studies on liner selection, inlet mode, and injection volume are suggested to improve method performance and reduce contamination. Due to its increased sensitivity, the optimised method is recommended for routine forensic-toxicology analyses in Kimia Malaysia.

Briefly, the direct injection GC-FID method developed in this study is a highly sensitive, precise, rapid, and reliable approach for the simultaneous determination of methanol and ethanol in local and foreign alcoholic beverages. However, certain limitations should be acknowledged. Direct injection of complex matrices may introduce interferences from congeners or sugars, while contamination and carry-over within the injection port can compromise reproducibility during successive runs. Future optimization strategies, such as improved inlet design, careful liner selection (e.g., deactivated split/splitless liners with wool packing), or the adoption of programmed temperature vaporizing (PTV) inlets, may help mitigate these issues. Additionally, simple pre-treatment steps, such as distillation or headspace sampling, could reduce matrix effects without significantly affecting throughput. Regular maintenance practices, including septum and liner replacement, are also recommended to enhance method robustness.

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