Chinese Journal of Analytical Chemistry 50.8 (2022) 100119

Contents lists available at ScienceDirect

Chinese Journal of Analytical Chemistry

journal homepage: www.elsevier.com/locate/cjac

Oil extracts from fresh and dried *Iban* ginger

Nurul Mirzha Syerliza Halimin[a], Mohammad Omar Abdullah[a,d,*], Noraziah Abdul Wahab[a], Radzuan Junin[b,c], Awang Ahmad Sallehin Awang Husaini[e], Augustine Agi[b]

[a] Department of Chemical Engineering & Energy Sustainability (ChemES), Faculty of Engineering, Universiti Malaysia Sarawak (UNIMAS), Kota Samarahan, Sarawak 94300, Malaysia
[b] Department of Petroleum Engineering, School of Chemical and Energy Engineering, Faculty of Engineering, Universiti Teknologi Malaysia, Johor Bahru 81310, Malaysia
[c] Institute for Oil and Gas (IFOG), Universiti Teknologi Malaysia, Johor Bahru 81310, Malaysia
[d] OSYIHM Farm, Balai Ringin, Serian, C/o OSYIHM Enterprise, Lot 8532, Muara Tabuan Land District, Kuching, Sarawak 93350, Malaysia
[e] Faculty of Resource Science and Technology, Universiti Malaysia Sarawak (UNIMAS), Kota Samarahan, Sarawak 94300, Malaysia

ARTICLE INFO

Keywords:
Plant essential oils
Iban ginger
Freshness
Oil yield
Phytochemical analysis
Chemical composition
Equivalent-extraction time

ABSTRACT

The present study aims to investigate the chemical properties of a new ginger species called *Iban* ginger. Oil extracts yield from both fresh and dried *Iban* ginger were compared via Soxhlet extraction production method. Subsequently, the chemical composition of the extracts was characterized and analysed. The associated chemical constituents and bioactive compounds were explored using gas chromatograph mass spectrometry (GCMS) and Fourier transformed infrared spectroscopy (FTIR) analysis for chemical constituents and plant active compound study. Results obtained show that yield of the oil increases with the increase of extraction time, freshness of ginger and type of solvent use. Although *Iban* ginger is known to be comparatively hotter in taste, the bioactive compounds properties are similar or in close agreement with other types of gingers reported in literature. Finally, acetone equivalent-extraction time of recycled ethanol is introduced herewith and found to be minimum around 2 h, as far as the present study is concerned.

1. Introduction

Ginger is known by its scientific name as *Zingiber officinale,* a member of Zingiberaceae family. Ginger can be used for food favouring, as ginger drink and can even as catalyst to enhance bioenergy production [1]. Over the years, ginger has become one of the herb plants that is often used for natural preservative and medicinal purpose due to the presence of its bioactive compounds that provides medicinal properties which helps to improve health. Natural preservatives derived from plant sources have been actively studied as an alternative to synthetic materials [2]. In the present research, while normal gingers are widely known and used by many, the local ginger *Iban* ginger is scarce and relatively expensive available locally in particular from Districts of Kuching and Serian, Sarawak, Malaysia. Unlike conventional gingers, the *Iban* ginger is known to be slightly hotter than ordinary gingers in taste and could enhance spicy sensation. Generally, the usage of organic manures improved quantity and quality of essential oil [3]. Organic manures play an important role in the growth and biomass of aromatic and medicinal plants leading to organic and cleaner production. Also, organic manures can improve chemical compositions and the quality of aromatic and medicinal plant viz. Dracocephalum kotschyi [4]. The present study reports the ginger oil extracts production from *Iban* ginger plant (*Zingiber officinale*) that are planted with sandy soils and organic chicken manures.

In the production of plant oil extracts, various solvents like methanol, ethanol, petroleum ether, dichloromethane and *n*-hexane had been widely used[1]. Alfaro et al. [5] observed that most researchers found ethanol as one of the best solvents due to high yield. Meanwhile, Gonçalves et al. [6] described the fractionation of Citrus bergamia essential oil using ethanol/water mixtures as solvents. Bio-oil production of Lemon Myrtle extract oils was conducted by Bakar et al. [7] in a fixed-bed reactor via pyrolysis method within a temperature range of 350–550 °C. It was found that an increase in pyrolysis temperature led to a decrease in organic acid and ketones. The present study opted for the affordable conventional Soxhlet distillation extraction technique together with the use of recycle solvent i.e. recycled ethanol to investigate its suitability to achieve good plant extract yield. Thus the primary aim of the present paper is to compare the *Iban* ginger extracts yield using acetone and ethanol, estimate the acetone equivalent-extraction time and effectiveness of employing recycled solvent i.e ethanol-recycled ethanol for synthesis of ginger oil as compared with acetone. It is to be noted that ethanol is about 30% more expensive than acetone. Apart from that, this paper also presents: (i) production of oil extracts derived from local

* Corresponding author.
E-mail address: amomar@unimas.my (M.O. Abdullah).

[1] It is to be noted that care must be taken when considering solvents like methanol and dichloromethane in extraction of products as these solvents are known toxic. There are usually for non-food applications.

https://doi.org/10.1016/j.cjac.2022.100119
Received 5 April 2022; Received in revised form 7 May 2022; Accepted 8 May 2022
Available online 12 May 2022

gingers from both fresh and dried ginger; and (ii) characterization and analysis of the *Iban* ginger extracts.

In term of synthesis methods, over the years, many methods had been employed by different researchers to extract different constituents from various types of gingers, each with their advantages and disadvantages. Mesomo et al. [8] reported the assessment of chemical profile and antibacterial activity of ginger roots extracts using supercritical CO_2 as solvent. While this method can give reliable yield and minimum decomposition changes, the overall process is expensive. Subsequently, Khanam [9] have also employed supercritical fluid extraction method but producing turmeric oil. Turmeric oil yield found through Soxhlet extraction was 5.954 wt% of turmeric powder whereas through supercritical fluid extraction, it varied from 2 to 5.3 wt%. Guo et al. [10] have conducted extraction of gingerols and shogaols from ginger (*Zingiber officinale roscoe)* through microwave-assisted extraction technique. Microwave extraction technique provide fast energy transfer thus rapid extraction and time saving. Yingngam and Brantner [11] also carried out solvent-free microwave extraction but combined with central composite design to boost the essential oil yield. Sasikala et al. [12] reported an ultrasound-assisted extraction and adsorption of polyphenols from Ginger Rhizome. Soxhlet extraction method is known to be marginally better method to extract essential oil from ginger as it gives good yield of ginger essential oil. However, the extraction conditions required the control of the parameters such as dryness of the ginger, operating temperature, length of extraction time and extraction solvent use all of which are crucial in the extraction process in order to obtain the desired compounds.

In general, there are many chemical constituents with more than 400 different compounds are found in ginger such as carbohydrates, lipids, terpenes and phenolic compounds, as reported by Prasad and Tyagi [13]. Ginger consist of two groups of chemical which are volatile oil that is consist of terpenes component and pungent compounds that consist of phenolic compound. Phenolic components in ginger consist of gingerol, shogaols and paradols, while terpene components of ginger are β-bisabolene, α-curcumene, zingiberene, α-farnesene and β-sesquiphellandrene [14]. Both terpenes and phenolic components are recognized as the main components of ginger extracts. Rhizome (root) is a medicinal part of ginger where it contains a lot of bioactive components that are associated with many health benefits and are widely used to treat many ailments. It has medicinal qualities where it helps to reduce inflammation, pain, nausea, lower cholesterol and also for digestion (Mao et al. [14]; dos Santos Reis et al. [15]).

2. Methodology

This section discusses on the sample preparation of ginger, extraction of ginger using Soxhlet method, separation of mixture and characterization of ginger extracts.

2.1. *Sample preparation of* Iban *ginger*

Iban ginger is selected as the raw material in this research study. The materials are purchased from local market in Serian Daily Market, Serian, Sarawak. The gingers are cleaned and washed in order to remove any extraneous matter. Next, the size of the gingers is reduced by cutting it into smaller slices to reduce the drying time before putting it inside the incubator oven for 1 day (24 h). The temperature of the oven is kept below 60 °C which is the suitable drying temperature for bark and root to avoid overheating which may result in an excessive loss of volatile components or the decomposition of chemical constituents of the ginger. The ginger is then grinded into powder form to increase the surface contact area, thereby improving the mass transfer of the chemical constituents from the ginger powder (tissue) into the solvent. The aim of powdering the ginger is also to break down the tissue and cell structure of the ginger in order to increase the contact exposure of the chemical constituents to the extracting solvent. Both the preparation of the sample for fresh and dried ginger are similar, except that the dried ginger undergoes a further 7-days of sun-drying. The sample preparation of the fresh ginger was conducted using 200 g of ginger, but after it was dried, the ginger only weighted around 60 g, approximately 70% weight reduction. Since the mass of sample used were constant throughout the experiments, viz. 15 g, the loss in weight of the fresh ginger after drying corresponding to around 35 g.

Table 1
Boiling point of solvents.

Solvent	Boiling point (°C)	Miscibility with H_2O	Threshold limit values (ppm)
Acetone	56	∞	1000
Ethanol	78	∞	1000

2.2. Lban *ginger oil extraction using Soxhlet method*

Soxhlet extraction method is an extraction technique which uses solvent to separate the essential oil from the raw material [16,17]. In this process, the Soxhlet extractor is connected to the condenser and round bottom flask. The extraction is performed using cellulose extraction thimble. The cellulose extraction thimble is then filled with 15 g of sample (*Iban* ginger powder) and placed in the Soxhlet extractor. Next, the round bottom flask that filled with 400 mL of extraction solvent which is acetone (C_3H_6O) (Merck; concentration <= 100%)) is heated, vaporized and condensed through the condenser. The condensed liquid is then in contact with the sample and the extract oil is obtained from the ginger rhizome. The production of a light yellow liquid indicates the presence of the extracts oils. As the overflow level is reached, the liquid is discharged back to the round bottom flask. The process is repeated until complete extraction at the specified extraction time is achieved. Throughout this process, the extraction temperature is remained constant at the boiling point of the acetone which is 56 °C. The duration of the extraction time is recorded at 1, 1.5 and 2 h during the process. The process is then repeated with the use of another solvent first with ethanol (C_2H_5OH) (Fisher Scientific; concentration = 95%), and subsequently recycled ethanol at 1.5 h of extraction. Two different solvents which are acetone and recycled ethanol are used in this study to compare the extraction efficiency between the two solvents and extraction schemes.

2.3. *Separation of mixture*

The mixture of the extract oil and solvent is separated by a simple distillation method. The mixture is heated based on the solvent boiling point for the collection of the extract oil. Table 1 shows the boiling point of the solvent used as the temperature of heating during the process in order to distil the solvent. In this process, the solvent inside the mixture is vaporized and passed through the condenser to be condensed and collected as a liquid in the beaker. The amount of the extract oil collected from fresh ginger and dried *Iban* ginger by using different solvents which are acetone and recycled ethanol are recorded and the yield of the extract oil is determined. The yield of the ginger extract oil can be expressed as [16]:

$$\text{Yield } (\%) = \frac{\text{mass of extracted oil (g)}}{\text{mass of sample taken (g)}} \text{ x } 100\ \%$$

2.4. *Sample analysis using gas chromatography mass spectrometry (GCMS) and Fourier transform infrared spectroscopy (FTIR)*

The characterization of ginger extract oil is performed in order to identify the chemical constituents in the ginger oil which are the presence of the bio-active compounds that is claimed to be an effective component that provides pharmacological properties in ginger. Analysis of chemical components in ginger extract oil is conducted using the application of gas chromatography - mass spectrometry (GCMS) analysis and

Fourier transform infrared spectroscopy (FTIR) analysis. GCMS is used to identify volatile components present in ginger extract oil and FTIR is used for determination of the functional groups of the ginger extract oil. This analysis is presented by comparing and matching of mass spectra and literature-based data.

The ginger oil is analyzed using Shimadzu (Japan) GC - QP2010 Plus gas chromatography equipped with a BPX-5 column with column dimension of 30 m × 0.25 mm i.d. × 0.25 μm, maximum temperature of 360/370 °C consisting of 5% phenyl polysilphenylene-siloxane. Helium (99.999%) at constant flow rate of 1 mL/min is used as the carrier gas. The initial oven temperature is programmed at 50 °C and held for 5 min and final temperature is programmed at 280 °C and held for 10 min. GC setup done in splitless injection mode. The injector and detector temperatures are 300 °C. Mass scan range (m/z) is conducted at 40–500 amu and the ion source temperature is 200 °C. This technique consists of the combination of GC which is used to separate the components of the mixture and MS where it gives information on the structural identification for each of the components [18]. Thus, the combination of GC and MS techniques give advantage for the analysis of qualitative and quantitative of volatile and semi volatile organic constituents in a mixture [19]. This analysis technique is widely used for determination of extract oil constituents and the evaluation of the components can be done by comparing the retention time or indices of the components passing through the column and their MS [20,21]. Thus, oils are classified by comparing their mass spectrum to the spectrometer database using the NIST147 mass spectral database and the chemical composition of the compounds are identified from the peak areas of GC.

Meanwhile, FTIR analyser model IRAffinity-1, Shimadzu spectrometer with the wave range in the region of 400 and 4000 cm^{-1} was used for spectrum analysis, to investigate the structure and functional group for the ginger oil. The ginger oil is placed at the sample holder in the FTIR spectrometer and run for the analysis. The wavelength of the scanning is set by using mid - IR spectrum region which is in the absorption range of 400 to 4000 cm^{-1} and scanned in transmittance vs wavenumber mode at room temperature.

3. Results and discussion

The present section discusses on the results of the findings and analysis of data from the experimental study conducted herein.

3.1. Effect of extraction time and ethanol solvent recycling on the yield of Iban ginger oil

Fig. 1 shows the effect of the extraction time on the yield of extract oil with acetone, ethanol and recycled ethanol as extracting solvent. The extraction time is varied in order to analyze the effect of the extraction time on the yield of the extract oil. The extraction processes

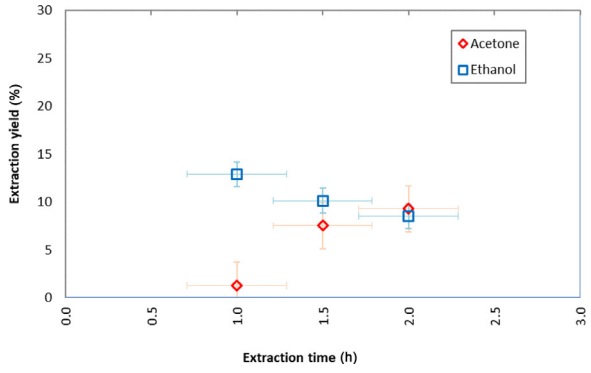

Fig. 1. Effect of extraction time on the yield of extract oil with different extraction solvent (Ethanol was recycled at 1.5 h of extraction).

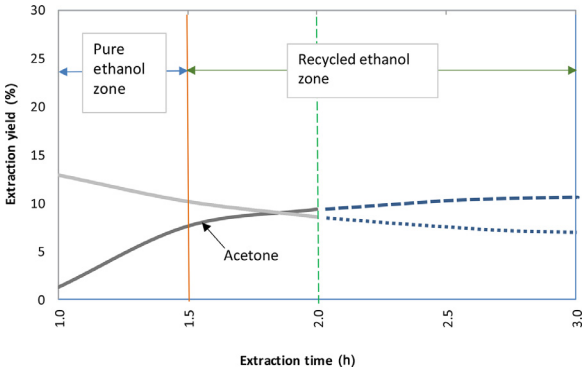

Fig. 2. The extraction yield of pure(initial) ethanol zone and recycled ethanol zone. Recycled ethanol started at 1.5 h and extracted yield was measured immediately thereafter.

are conducted at three time intervals i.e. for 1, 1.5 and 2 h. The recovered ethanol from the distillation process is being recycled at period of 1.5 h. Based on Fig. 1, for acetone solvent, the ginger oil yield is 1.27%, 7.52% and 9.27% at extraction time of 1, 1.5 and 2 h, respectively. This is also corresponding to the previous studies which explained that the longer extraction period will normally resulted in a higher percentage yield due to longer period of contact between the solvent and the solute, where the longer contact time resulted in more mass transfer by the system [22]. In the case of ethanol solvent, the use of ethanol resulted in a higher percentage yield of 12.91% initially. This follow by a gradual reduction of yield to 10.14% and 8.52%, after 1.5 and 2.0 h of extraction time, respectively. The different of the results obtained is attributed to the scheme of the extraction in which the solvent collected or recovered from the distillation process is being used again during the extraction process. Ethanol is used during the initial stage up to 1.5 h followed by recycled ethanol thereafter. Acetone is used for extract oil extraction during the entire process of 3 min (Fig. 2). The use of recycled solvent (recycle ethanol) made the extraction process reduced in effectiveness which in turn leading to decreasing yield results. Nevertheless, the practical yield for the present study can up to 2 h in which the yield generated is equal to that of pure acetone.

3.2. Effect of dryness on the amount of extract oil obtained and the yield

The results of the extraction yield are shown in Fig. 3. Based on this figure, the yield of the oil extracted from the fresh ginger gave the percentage yield of 1.27% when acetone is used as a solvent for extraction and 12.91% when recycled ethanol is used as a solvent for extraction. While, the percentage yield of dried ginger extracted using acetone and recycled ethanol is 1.24% and 4.13% respectively. The observation shows that the oil extracted from fresh ginger is higher compared to the oil extracted from dried ginger.

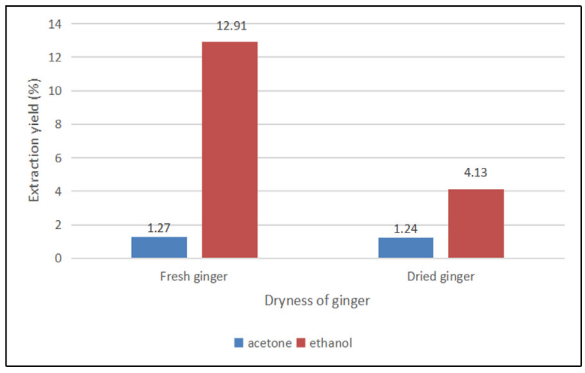

Fig. 3. Effect of dryness on the yield of extract oil.

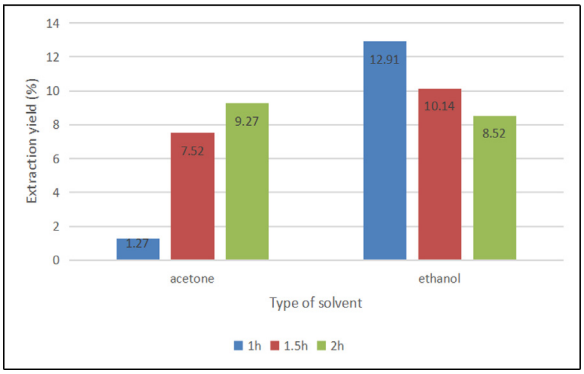

Fig. 4. Effect of solvent type (acetone and ethanol-recycled ethanol) on the yield of extract oil.

This observation is in line with the findings of Mirhosseini et al. [23] who reported the treatment of raw material by sun drying resulted in faster vaporization process which in turn causing decrease of the oil content. Thus, leads to the reduction of the extract oil yield. It is also correlated with the finding of Arabhosseini et al. [24] and Mahayothee et al. [25] who found the oil content of the raw materials will decrease with the increasing of drying temperature and from a longer drying time. This shows that the obtained result of the oil can be influenced by the drying conditions where higher temperature and longer drying time induce the loss of extract oil. Nevertheless, according to Mahayothee et al. [25] even though volatile oil contents reduced after drying due to different drying methods and drying rates but their compositions were unchanged.

3.3. Effect of solvent and extraction scheme on the yield of extract oil

In this study, two solvents are used for comparison which is recycled ethanol and acetone. The data obtained referring to the yield of oil produced using recycled ethanol and acetone is presented in Fig. 4. Extraction using acetone which is conducted at 1hr gives extraction yield of 1.27% whereas using recycled ethanol gives extraction yield of 12.91%. For the extraction process which is carried out at 1.5 h using recycled ethanol gives 10.14% of the extraction yield while 7.52% is obtained from the extraction using acetone. Both of this results reveals that recycled ethanol can yield more oil compared to acetone, as far as present study is concerned. It was also observed that the yield of oil extracted using recycled ethanol which is conducted at 2 h was 8.52%. While 9.27% is obtained from the extraction using acetone which is higher as compared to recycled ethanol.

Based on the overall results, the extraction of oil using acetone resulted in the lower percentage yields as compared to recycled ethanol at extraction time up to 2 h. The difference in the results obtained could be due to the extraction procedure and the polarity of the solvents. Generally, ethanol is more polar than acetone [26,27]. These results are in good agreement with literature. According to Truong et al. [27], extraction efficiency favours higher polarity solvents where extraction using methanol, distilled water and ethanol resulted in higher extraction yields compared to chloroform, dichloromethane and acetone. Therefore, in the present study, initially finding indicates that ethanol is the better solvent to extract oil from the *Iban* ginger since it resulted in superior extraction yield compared to acetone. However, the use of recycle ethanol would be pragmatic only at the initial time interval up to 2 h; at 2 h and beyond, acetone will outperform recycle ethanol, as far as present study is concerned.

3.4. Analysis and characterization of ginger extract oil

The analysis was carried out by GCMS and FTIR spectrometer. The results are presented and discussed in Sections 3.4.1 and 3.4.2, respectively.

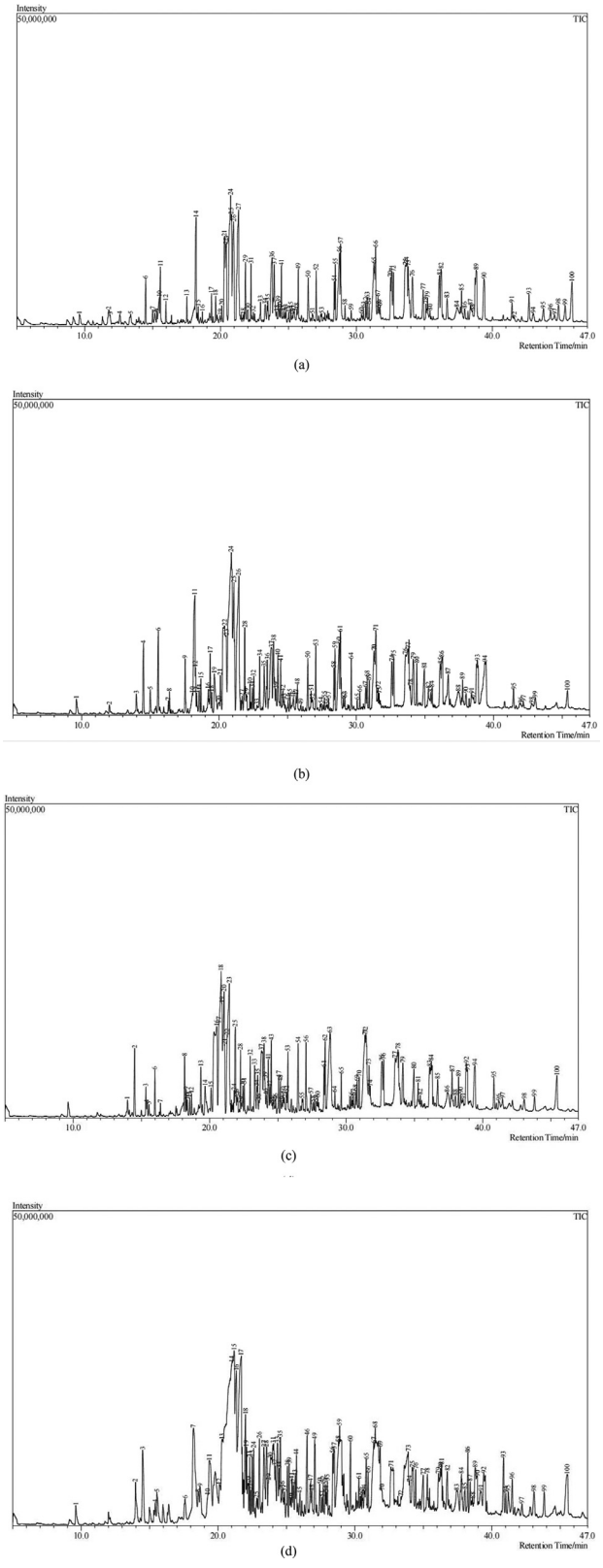

Fig. 5. GCMS chromatogram at extraction time of 1 h. (a) Fresh *Iban* ginger + acetone; (b) fresh *Iban* ginger + ethanol; (c) dried *Iban* ginger + acetone; (d) dried *Iban* ginger + ethanol.

3.4.1. Analysis of ginger extract oil using gas chromatography mass spectrometry (GCMS)

Fig. 5a represents the GCMS chromatogram of fresh *Iban* ginger where acetone is used as the extraction solvent while Fig. 5b

Table 2
GCMS analysis of chemical constituents of fresh *Iban* ginger using different solvent type.

Compounds	Chemical formula	Acetone			Ethanol		
		peak	Retention time (min)	Amount (%)	peak	Retention time (min)	Amount (%)
Zingerone	$C_{11}H_{14}O_3$	36	23.796	3.17	37	23.812	3.14
α-farnesene	$C_{15}H_{24}$	25	20.811	2.60	24	20.902	12.18
β-bisabolene	$C_{15}H_{24}$	26	20.959	4.56	25	21.093	4.05
β-sesquiphellandrene	$C_{15}H_{24}$	27	21.330	6.27	26	21.453	6.39
Zingiberene	$C_{15}H_{24}$	24	20.736	6.32	46	25.420	0.52
Decanal	$C_{10}H_{20}O$	6	14.487	0.86	4	14.500	1.20
[6]-Gingerol	$C_{17}H_{26}O_4$	72	32.721	1.00	75	32.736	1.00
[6] - Shogaol	$C_{17}H_{24}O_3$	73	33.600	4.72	76	33.595	4.93
					77	33.787	
		74	33.710				
α-Curcumene	$C_{15}H_{22}$	22	20.390	1.21	23	20.530	1.76
		23	20.433				
Geranyl acetate	$C_{12}H_{20}O_2$	14	18.197	2.48	11	18.248	3.54
Geraniol	$C_{10}H_{18}$	11	15.567	1.06	6	15.584	1.37

Table 3
GCMS analysis of chemical constituents of dried *Iban* ginger employing different solvent type.

Compounds	Chemical formula	Acetone			Ethanol		
		peak	Retention time (min)	Amount (%)	peak	Retention time (min)	Amount (%)
Zingerone	$C_{11}H_{14}O_3$	37	23.799	3.69	30	23.835	2.30
α-Farnesene	$C_{15}H_{24}$	19	20.905	1.05	18	22.009	1.69
β-Bisabolene	$C_{15}H_{24}$	20	21.058	4.64	16	21.348	1.41
β-Sesquiphellandrene	$C_{15}H_{24}$	23	21.435	5.88	17	21.708	4.64
Zingiberene	$C_{15}H_{24}$	17	20.675	9.13	9	18.689	0.71
		18	20.841				
Decanal	$C_{10}H_{20}O$	2	14.503	1.38	-	-	-
[6]-Gingerol	$C_{17}H_{26}O_4$	76	32.747	1.00	76	34.441	1.43
					77	34.996	
[6] - Shogaol	$C_{17}H_{24}O_3$	77	33.590	5.70	71	32.638	2.26
		78	33.795				
α-Curcumene	$C_{15}H_{22}$	16	20.507	1.09	22	22.393	0.25
Geranyl acetate	$C_{12}H_{20}O_2$	8	18.161	0.96	7	18.204	4.14
Geraniol	$C_{10}H_{18}$	5	15.542	0.20	5	15.542	0.46

represents GCMS chromatogram of fresh *Iban* ginger where ethanol is used as a solvent for the extraction. Next, Fig. 5c and d shows GCMS chromatogram of dried *Iban* ginger in which acetone and ethanol are used as the solvent for the extraction process. Some of the major compounds identified in fresh and dried ginger are listed in Tables 2 and 3 along with their peak, retention time as well as their percentage compositions for the comparison. The main constituents of the *Iban* ginger oil in the terpene compounds is α-farnesene, β-bisabolene, β-sesquiphellandrene, zingiberene and α-curcumene. In this research, all of this components are present in both fresh and dried *Iban* ginger. Next, the results also show that the major constituents of phenolic compounds are also found in ginger oil which are zingerone, shogaols and gingerols.

As can be seen in Table 2 for GCMS analysis of chemical constituents of fresh *Iban* ginger oil when acetone is used as solvent, the sample is found to have zingerone (3.17%), α-farnesene (2.60%), β-bisabolene (4.56%), β-sesquiphellandrene (6.27%), zingiberene (6.32%), decanal (0.86%), [6]-gingerol (1.00%), [6]-shogaol (4.27%), α-curcumene (1.21%), geranyl acetate (2.48%) and geraniol (1.06%) as the most abundant compounds. According to the results obtained from the studies, the major component present in this sample is zingiberene followed by β-sesquiphellandrene and [6]-shogaol. These findings are also correlated with the report of the previous studies in which the researcher stated that the main compounds found in the fresh ginger oil are zingiberene, α-farnesene, β-sesquiphellandrene and α-curcumene (see Mahboubi [28]). While for ethanol, the amount of the main compounds found in the fresh ginger oil are zingerone (3.14%), α-farnesene (12.18%), β-bisabolene (4.05%), β-sesquiphellandrene (6.39%), zingiberene (0.52%), decanal (1.20%), [6]-gingerol (1.00%), [6] - shogaol (4.93%), α-curcumene (1.76%), geranyl acetate (3.54%) and geraniol (1.37%). α-farnesene is the main component contained in this sample followed by β-sesquiphellandrene and zingiberene. These results are also in harmony with the findings of Mahboubi [28] as have been explained previously.

On the other hand, GCMS analysis of dried *Iban* ginger for acetone as shown in Table 3 showed that the most abundant compounds are zingerone (3.69%), α-farnesene (1.05%), β-bisabolene (4.64%), β-sesquiphellandrene (5.88%), zingiberene (9.13%), decanal (1.38%), [6]-gingerol (1.00%), [6]-shogaol (5.70%), α-curcumene (1.09%), geranyl acetate (0.96%) and geraniol (0.20%). This finding show that zingiberene, β-sesquiphellandrene and [6]-shogaol are the main components present in the sample. Furthermore, zingerone (2.30%), α-farnesene (1.69%), β-bisabolene (1.41%), β-sesquiphellandrene (4.64%), zingiberene (0.71%), [6]-gingerol (1.43%), [6]-shogaol (2.26%), α-curcumene (0.25%), geranyl acetate (4.14%) and geraniol (0.46%) are the major compounds found in dried *Iban* ginger oil for ethanol as shown in Table 3. Besides, there is no decanal compounds is detected in this sample. β-sesquiphellandrene is the main component contained in this dried ginger oil followed by geranyl acetate and zingerone.

Based on these studies, the largest amount of zingerone and shogaols are found in dried ginger oil as compared to fresh ginger. Both zingerone and shogaols are one of the pungent compounds of the ginger oil. Zingerone is mainly occurred in dry ginger. Ahmad et al. [29] and another earliest study by Connell and Sutherland [30] also stated that large amount of zingerone and shogaols are found in dried ginger whilst only small amounts are discovered in fresh ginger. The presence of these compounds in the ginger oil gives antioxidant and anti-inflammatory properties. Moreover, gingerols as one of the main bioactive compounds are

found in the small amount in both fresh and dried *Iban* ginger which is only around 1.00%–1.43%. These compounds also provide medicinal properties where it also gives anti-inflammatory and antioxidant effects as zingerone and shogaols. The reason that causes low gingerol content that is detected in the ginger can be that gingerols have been decomposed into zingerone and shogaol at high temperature of more than 45 °C. [31]. Thus, also explained the reason for higher amount of zingerone and shogaols in the samples. For terpenes compounds, the most abundant found in fresh ginger oil is zingiberene and α-farnesene (Table 2) while zingiberene and β-sesquiphellandrene in dried ginger oil (Table 3). In addition, acetone extract contains substantially more zingiberene than ethanol extract in both fresh and dried ginger. Zingiberene is one of the main constituent of Iban ginger oil in terpenes compound. The previous studies by Gonzalez-Burgos and Gomez-Serranillos [32] show that terpenes have been shown to have antimicrobial, anti-inflammatory, antiulcerogenic, anticancer and antioxidant properties. Terpenes have a chemical structures that indicate antioxidant activities, which appear to be an important molecules with high antioxidant potential for human health [32]. According to Borges et al. [33], acetone was the most effective extraction solvent in terms of antioxidant capability and there was no extraction method excelled at extracting antioxidant compounds as it was highly depended on the extraction solvent used. Earlier, Sharma and Gupta [34] also stated that the traditional extraction method involves juicing and introducing acetone as the extraction solvent is more effective than ethanol. Thus, explained the reason for the higher amount of zingiberene in both fresh and dried ginger when using acetone as the extraction solvent compared to using ethanol as an extraction solvent. The results show that the chemical composition of the ginger oil is found varied among the samples. Nonetheless, all these compounds poses similar function where it contributes to pharmacological properties of ginger. Lastly, the presence of geranyl acetate gives floral fruity and sweet aroma of the ginger.

3.4.2. Analysis of ginger extract oil using Fourier transform infrared (FTIR) spectrometer

The infrared spectrum of fresh *Iban* ginger oil using acetone as an extraction solvent are presented in Fig. 6a. The wavelength of the infrared spectrum is in the range of 561.29 cm^{-1} to 3379.29 cm^{-1}. There are 10 functional compounds found in the sample. The spectrum of ginger oil showed an OH–H group at 3379.29 cm^{-1}. This broad absorption band confirms the existence of hydroxyl compounds as it followed with presence of spectra at the frequencies of 1369.46 cm^{-1}, 1031.92 cm^{-1} and 806.25 cm^{-1}. Thus, this shows that *Iban* ginger contains phenolic compounds which possess antioxidant potential which are zingerone, [6]-shogaols and [6]-gingerols. Next, the presence of narrow band at 2924.09 cm^{-1} can be attributed to aliphatic compounds which is identified as carboxylic acid group. This finding are supported by the previous studies which also found that at wavelength between 2872 cm^{-1} and 3022 cm^{-1} are confirmed to be related to carboxylic acid group Farshbaf-Sadigh [35]. It is also correlated with the finding of Jayanudin and Rochmadi [36] who reported that the band appeared at 2924.7 cm^{-1} in the spectrum of ginger oleoresin are identified as an OH-H group. The presence of this carboxylic acid compounds may be attributed to fatty acids contents in ginger oil. Other than that, there are peaks appeared at 1514.12 cm^{-1} and 1448.54 cm^{-1} illustrated the presence of C=C aromatic compounds with the vibration of ring aromatic stretch. This band possibly confirms the existence of zingeberene and bisabolene compounds in the ginger. These results are in line with the findings of Purnomo et al. [37] which reported that the presence of C=C aromatic compounds are observed at 1514.98 cm^{-1} and 1449.41 cm^{-1}. The wavelength of 1707 cm^{-1} with intensity of 83.31 is also observed where it indicated as ester group. This shows that monoterpene compounds such as geranyl acetate may be found in the ginger oil. Peak 1707 cm^{-1} may also confirms the existence of ketones such as monoterpene compounds and aldehyde such as decanal

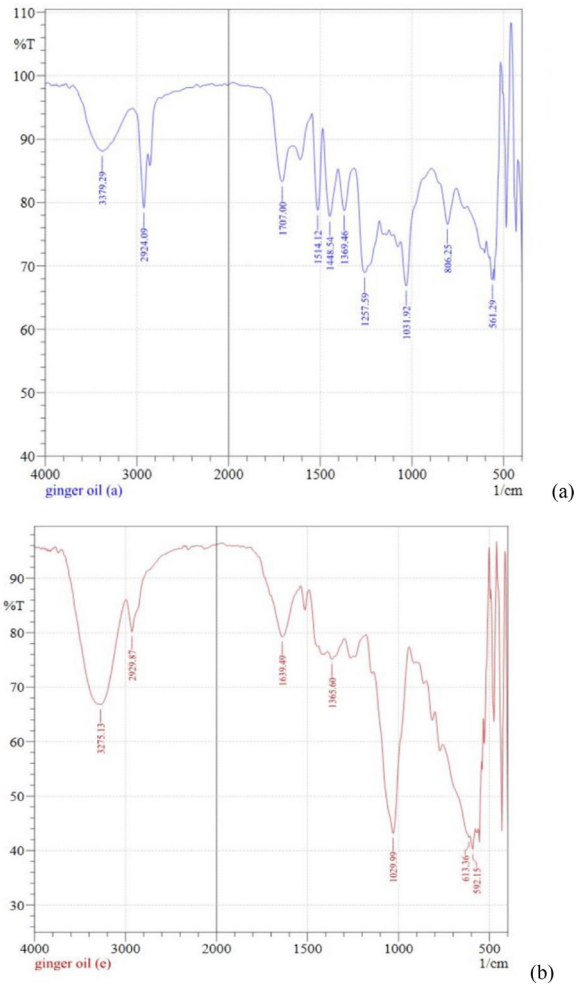

Fig. 6. Infrared spectrum of (a) fresh *Iban* ginger extract oil using acetone as an extraction solvent; and (b) fresh *Iban* ginger extract oil using ethanol as an extraction solvent.

and geranial compounds in the ginger oil. Other bands are also observed at 1257.59 cm^{-1}, 806.25 cm^{-1} and 561.29 cm^{-1}.

Next, Fig. 6a shows the infrared spectrum of fresh *Iban* ginger oil using ethanol as an extraction solvent. The wavelength of the infrared spectrum are in the range of 592.15 cm^{-1} to 3275.13 cm^{-1}. There are 7 functional compounds are found in the sample. Based on Fig. 6b, the FTIR spectra of this ginger oil extracted using ethanol at 3275.13 cm^{-1} is indicated as hydrogen bond. The presence of this band is confirmed to be related to hydroxyl compounds with the presence of the spectra at the frequencies of 1365.6 cm^{-1}, 1029.99 cm^{-1} and 613.36 cm^{-1}. This indicates that *Iban* ginger may contain phenolic compounds which have antioxidant potential as also stated in Fig. 6a. The presence of phenolic compounds may be attributed to zingerone, [6]-shogaols and [6]-gingerols. The narrow band appeared at 2929.87 cm^{-1} illustrated the presence of aliphatic compounds. Farshbaf-Sadigh et al. [35] reported that the presence of absorption in the spectrum of between 2872 cm^{-1} and 3022 cm^{-1} were determined to be associated with carboxylic acid group. (Farshbaf-Sadigh et al. [35]). Thus, this band is identified as a carboxylic acid group and this may be due to fatty acids contents in ginger oil. The wavelength of 1639.49 cm^{-1} with intensity of 79.22 is also observed where it indicated the presence of C=C aromatic compound. This band confirms the existence of zingeberene and bisabolene compounds in the ginger. Other band is also identified at 592.15 cm^{-1}. Based on the discussion of the infrared spectrum of the fresh *Iban* ginger oil in both Fig. 6a and b, it can be deduced that both samples pos-

sess similar functional compounds. This also shows that the results are not really affected by the extraction solvents. From the FTIR analysis, the organic compounds that are able to be identified are phenolic compounds, carboxylic acid group, aldehydes, ketones, esters and aromatic compounds.

Finally, for completeness purpose it is worth to be noted that, in term of extract oil quality, it was reported generally the usage of organic manures improved quantity and quality of extract oil [4]. The present study reported the extract oil production from *Iban* ginger plant (*Zingiber officinale*) that are planted with sandy soils and organic chicken manures.

4. Conclusion and recommendation for the further work

The present work extract oil production from both fresh and dried *Iban* ginger via Soxhlet extraction production method. It also describes solvent design schemes and introduces acetone equivalent-extraction time concept in which recycled ethanol has been shown can be feasible for use here for the *Iban* ginger plant oil extraction. In this work, from the Soxhlet extraction experiment conducted, on the effect of dryness shows that the oil yield extracted from fresh ginger is higher compared to the oil extracted from dried ginger. Ethanol is superior solvent compared to acetone for extracting bioactive compounds from ginger since it resulted in higher yield of ginger oil. Recycled ethanol extraction scheme had been proven herein for practical use especially during ~2 h of acetone equivalent-extraction time, as far as the present study is concern. Based on the analysis results from GCMS and FTIR, it is further concluded that the components present in *Iban* ginger oil offers properties close or similar to other gingers such as red ginger. The organic compounds identified are phenolic compounds, carboxylic acid group, aldehydes, ketones, esters and aromatic compounds. The main constituents of ginger oil in the terpene compounds are α-farnesene, β-bisabolene, β-sesquiphellandrene, zingiberene and α-curcumene and major constituents of phenolic compounds which are zingerone, shogaols and gingerols found in both fresh and dried *Iban* ginger. The present findings may pave the way for further research on the bioactive compounds and the more detail extracts oil properties for various applications e.g. insect repellent, soap, detergent, and for external usages of various treatments in the near future. Also, the effects of temperature can be further tested in the future.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

The present works are partly supported by the Ministry of Higher Education, Malaysia under FRGS Grant Scheme(FRGS/1/2016/TK07/UNIMAS/01/2). The authors also grateful to OSYIHM Enterprise for collaboration and assistance for providing some farming materials. We appreciate the assistance of Ms. Nur Farahiyah in this project. The authors acknowledge UNIMAS for providing research facilities. And, we are indebted to all staff and friends for their continuous supports throughout this project.

References

[1] John M, Abdullah MO, Hua TY, Nolasco-Hipólito C. Techno-economical and energy analysis of sunflower oil biodiesel synthesis assisted with waste ginger leaves derived catalysts. Renew Energy 2021;168:815–28.

[2] Marzlan AA, Muhialdin BJ, Abedin NHZ, Mohammed NK, Abadl MMT, Roby BHM, Hussin ASM. Optimized supercritical CO_2 extraction conditions on yield and quality of torch ginger (Etlingera elatior (Jack) RM Smith) inflorescence essential oil. Ind Crops Prod 2020;154:112581.

[3] Fallah S, Rostaei M, Lorigooini Z, Surki AA. Chemical compositions of essential oil and antioxidant activity of dragonhead (Dracocephalum moldavica) in sole crop and dragonhead-soybean (Glycine max) intercropping system under organic manure and chemical fertilizers. Ind Crops Prod 2018;115:158–65.

[4] Fallah S, Mouguee S, Rostaei M, Adavi Z, Lorigooini Z, Shahbazi E. Productivity and essential oil quality of Dracocephalum kotschyi under organic and chemical fertilization conditions. J Clean Prod 2020;255:120189 2020 May 10.

[5] Alfaro MJ, Bélanger JM, Padilla FC, Paré JJ. Influence of solvent, matrix dielectric properties, and applied power on the liquid-phase microwave-assisted processes (MAP™) extraction of ginger (Zingiber officinale). Food Res Int 2003;36(5):499–504.

[6] Gonçalves D, Koshima CC, Batista FRM, da Costa Rodrigues CE. Performance of continuous countercurrent extractor on the fractionation of citrus bergamia essential oil using ethanol/water mixtures as solvents. Chem Eng Res Des 2018;137:566–76.

[7] Bakar MSA, Ahmed A, Jeffery DM, Hidayat S, Sukri RS, Mahlia TMI, Jamil F, Khurrum MS, Inayat A, Moogi S, Park YK. Pyrolysis of solid waste residues from Lemon Myrtle essential oils extraction for bio-oil production. Bioresour Technol 2020:123913.

[8] Mesomo MC, Corazza ML, Ndiaye PM, Dalla Santa OR, Cardozo L, de Paula Scheer A. Supercritical CO_2 extracts and essential oil of ginger (Zingiber officinale R.): chemical composition and antibacterial activity. J Supercrit Fluids 2013;80:44–9.

[9] Khanam S. Influence of operating parameters on supercritical fluid extraction of essential oil from turmeric root. J Clean Prod 2018;188:816–24.

[10] Guo JB, Fan Y, Zhang WJ, Wu H, Du LM, Chang YX. Extraction of gingerols and shogaols from ginger (Zingiber officinale Roscoe) through microwave technique using ionic liquids. J Food Compos Anal 2017;62:35–42.

[11] Yingngam B, Brantner A. Boosting the essential oil yield from the rhizomes of cassumunar ginger by an eco-friendly solvent-free microwave extraction combined with central composite design. J Essent Oil Res 2018;30(6):409–20.

[12] Sasikala P, Chandralekha A, Chaurasiya RS, Chandrasekhar J, Raghavarao KSMS. Ultrasound-assisted extraction and adsorption of polyphenols from ginger rhizome (Zingiber officinale). Sep Sci Technol 2018;53(3):439–48.

[13] Prasad Sahdeo, Tyagi Amit K. Ginger and its constituents: role in prevention and treatment of gastrointestinal cancer. Gastroenterol. Res. Pract. 2015;Article ID 142979. doi:10.1155/2015/142979.

[14] Mao QQ, Xu XY, Cao SY, Gan RY, Corke H, Beta T, Li HB. Bioactive compounds and bioactivities of ginger (*Zingiber officinale* Roscoe). Foods 2019;8(6):185.

[15] dos Santos Reis N, de Santana NB, de Carvalho Tavares IM, Lessa OA, dos Santos LR, Pereira NE, Soares GA, Oliveira RA, Oliveira JR, Franco M. Enzyme extraction by lab-scale hydrodistillation of ginger essential oil (Zingiber officinale Roscoe): Chromatographic and micromorphological analyses. Ind Crops Prod 2020;146:112210.

[16] Baser KHC, Buchbauer G. Handbook of essential oils: science, technology, and applications. CRC press; 2015.

[17] Ferrentino G, Morozova K, Horn C, Scampicchio M. Extraction of essential oils from medicinal plants and their utilization as food antioxidants. Curr Pharm Des 2020;26(5):519–41.

[18] Sparkman OD, Penton Z, Kitson FG. Gas Chromatography and Mass Spectrometry: A Practical Guide. Academic Press; 2011.

[19] Sneddon J, Masuram S, Richert JC. Gas chromatography-mass spectrometry-basic principles, instrumentation and selected applications for detection of organic compounds. Anal Lett 2007;40(6):1003–12.

[20] Abdullahi A, Khairulmazmi A, Yasmeen S, Ismail IS, Norhayu A, Sulaiman MR, Ahmed OH, Ismail MR. Phytochemical profiling and antimicrobial activity of ginger (Zingiber officinale) essential oils against important phytopathogens. Arab J Chem 2020;13(11):8012–25.

[21] Kıvrak Ş. Essential oil composition and antioxidant activities of eight cultivars of lavender and lavandin from Western Anatolia. Ind Crops Prod 2018;117:88–96.

[22] Baldosano HY, Beatriz M, Castillo MG, Elloran CDH, Bacani FT. Effect of particle size, solvent and extraction time on tannin extract from Spondias purpurea bark through Soxhlet extraction. In: Proceedings of the *DLSU research congress*, 3; 2015. p. 1–6.

[23] Mirhosseini F, Rahimmalek M, Pirbalouti AG, Taghipoor M. Effect of different drying treatments on essential oil yield, composition and color characteristics of Kelussia odoratissimaMozaff. J Essent Oil Res 2015;27(3):204–11.

[24] Arabhosseini A, Padhye S, van Beek TA, van Boxtel AJ, Huisman W, Posthumus MA, Muller J. Loss of essential oil of tarragon (Artemisia dracunculus L.) due to drying. J Sci Food Agric 2006;86(15):2543–50.

[25] Mahayothee B, Thamsala T, Khuwijitjaru P, Janjai S. Effect of drying temperature and drying method on drying rate and bioactive compounds in cassumunar ginger (Zingiber montanum). J Appl Res Med Aromat Plants 2020:100262.

[26] Chaaban A, Santos VMCS, Martins CEN, Brum JS, Bertoldi FC, Molento MB. Tissue damage and cytotoxic effects of tagetes minuta essential oil against Lucilia cuprina. Exp Parasitol 2019;198:46–52.

[27] Truong DH, Nguyen DH, Ta NTA, Bui AV, Do TH, Nguyen HC. Evaluation of the use of different solvents for phytochemical constituents, antioxidants, and *in vitro* anti-inflammatory activities of severinia buxifolia. J Food Qual 2019;2019:1–9.

[28] Mahboubi M. Zingiber officinale Rosc. essential oil, a review on its composition and bioactivity. Clin Phytosci 2019;5(6):1–12.

[29] Ahmad B, Rehman MU, Amin I, Arif A, Rasool S, Bhat SA, Afzal I, Hussain I, Bilal S, & Rahman Mir M. A review on pharmacological properties of zingerone (4-(4-Hydroxy-3- methoxyphenyl)-2-butanone). Sci World J 2015:1–6.

[30] Connell D, Sutherland M. A re-examination of gingerol, shogaol and zingerone, the pungent principles of ginger (Zingiber officinale Roscoe). Aust J Chem 1969;22(5):1033–43.

[31] Gaedcke, F., and Feistel, B. Ginger *extract preparation.* U.S. Patent Application 10/496,885, filed February 10, 2005.

[32] Gonzalez-Burgos E &, Gomez-Serranillos MP. Terpene compounds in nature: a review of their potential antioxidant activity. Curr Med Chem 2012;19(31):5319–41.
[33] Borges A, Jose H, Homem V, Simoes M. Comparison of techniques and solvents on the microbial and antioxidant potential of extracts from acacia dealbata and olea europaea. Antibiotics 2020;9(2):48.
[34] Sharma SS, Gupta YK. Reversal of cisplatin-induced delay in gastric emptying rats by ginger (Zingiber officinale). J Ethnopharmacol 1998;62(1):49–55.
[35] Farshbaf-Sadigh A, Jafarizadeh-Malmiri H, Anarjan N, Najian Y. Preparation of ginger oil in water nanoemulsion using phase inversion composition technique: effects of stirring and water addition rates on their physico-chemical properties and stability. Z Phys Chem 2019:1–20.
[36] Jayanudin, Rochmadi. Encapsulation of red ginger oleoresin (Zingiber officinale var rubrum) with chitosan as wall material. Int J Pharm Pharm Sci 2017;9(8):1–6.
[37] Purnomo H, Jaya F, Widjanarko SB. The effects of type and time of thermal processing on ginger (Zingiber officinale Roscoe) rhizome antioxidant compounds and its quality. Int Food Res J 2010;17:335–47.