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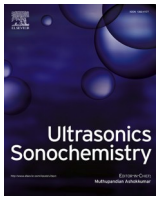

Ultrasonic-assisted extraction to enhance the recovery of bioactive phenolic compounds from *Commiphora gileadensis* leaves

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ABSTRACT

The "ultrasonic-assisted extraction (UAE)" method was utilized in this work to assess how different process parameters affected the yield and recovery of phenolic compounds from the leaf of *Commiphora gileadensis*, which is one of the medicinal plants with a variety of biological functions. Its leaf is used for a various of therapeutic applications, such as the treatment of bacterial infections, inflammation, and wound healing. The "One-Factor-At-a-Time (OFAT)" approach was employed to examine the impacts of various UAE process parameters on the process of extraction, which include time of extraction, sample/solvent ratio, ultrasonic frequency, and solvent (ethanol) concentration. The extracts were then investigated for the presence of several phytochemicals using analytical techniques such as "Gas Chromatography-Mass Spectroscopy (GC–MS)" and "Fourier Transform Infrared Spectroscopy (FTIR)" studies. The findings showed that the maximum extraction yield, the total phenolic content (TPC), and the total flavonoids content (TFC) of the ethanolic extract of the leaves of *C. gileadensis* using the UAE method were at 31.80 ± 0.41 %, 96.55 ± 2.81 mg GAE/g d.w. and 31.66 ± 2.01 mg QE/g d.w. accordingly under a procedure duration of 15 min, ultrasonic frequency of 20 kHz, solvent/sample ratio of 1:20 g/mL, and solvent concentration of 40 % v/v. The leaves extract of *C. gileadensis* included 25 phenolic compounds that were previously unreported, and GC–MS analysis confirmed their presence. Hence, it follows that the UAE technique can successfully extract the phytochemicals from *C. gileadensis* for a variety of therapeutic uses.

1. Introduction

The use of medicinal plants to cure and prevent a wide range of illnesses and disorders has been widespread since antiquity. In today's developing nations, safeguarding herbal products and therapeutic plants and preserving their effectiveness and quality are of highest importance [1]. Approximately eighty percentage of the world's population already benefits from the products of herbals in some way, in accordance with the World Health Organization (WHO), whether as dietary supplements or alternative medicines for a range of medical issue [2,3]. Despite the large number of identified plants utilized for alternative medicine, there are still quite a few that are yet to be found, underscoring the necessity to find new medicinal plants for sustainability [4].

One of these plants is the Burseraceae family tree *Commiphora gileadensis* [5–7]. It came from the Arabian Peninsula's southern Kingdom of Sheba [8–10] although it has also been observed in other places, including Oman, Yemen, Somalia, Sudan, and Ethiopia [11–13]. *C. gileadensis* is recognized for its opulent fragrance and several health advantages coming from its leaves, bark, seeds, wood, and sap [12,14]. Also, known as besham or becham in the region of the Middle East, it's is used in herbal remedies [5,15]. *C. gileadensis* has been utilized historically and is still used now in several Middle Eastern nations as an substitute remedy for several kinds of illnesses [9,16,17]. It has medicinal properties for treating ailments like, stomach troubles urinary retention, liver disorders, constipation, headaches, and jaundice and has shown promise in treating ~~a variety of conditions, including~~ certain types of cancer cell lines [5,14].

The plant's aerial portions have undergone phytochemical examination, which uncovered the existence of phenolic, saponins, flavonoids, sterols, and triterpenes [18,19]. Moreover, the plant has been discovered which contains antimicrobial qualities [17]. It acts as a diuretic, cancer analgesic, and is utilized in traditional Arabic medicine in many

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African nations to treat opportunistic infections [17,20].

Various extraction methods, both standard and unconventional, are applied to extract bioactive components from plant matrix. Different techniques and solvents may be used to recover phenolics from plant samples depending on their kind and distribution. Due to its great efficacy, low time and solvent needs, and compatibility for thermally sensitive chemicals, ultrasonic-assisted extraction (UAE) is an attractive technique for recovering plant bioactive components [21,22]. It additionally serves as a well-known eco-friendly extraction technique [23]. UAE is a fascinating alternative to traditional techniques because it eliminates the degradation of thermosensitive compounds, reduces the need for solvents, saves energy, and shortens extraction times for a greater yield [24]. The food industry, where it stimulates chemical reactions through cavitation, has recognised the efficiency of ultrasonic technology [23,25]. Acoustic cavitation, in which ultrasound vibrations create compression and rarefaction in an environment and result in inner changes in structure in food matrices, is the fundamental idea of UAE [26–28]. Sonication can be used for neither indirect or direct UAE, in which the effects of the ultrasound act on the sample directly or indirectly through a medium [21,29]. Direct sonication is preferred for small-scale extractions where an ultrasonic probe is usually utilized, while indirect ultrasound in ultrasound reactors or baths is used for larger-scale extractions [30–32]. The UAE method is essential for enhancing phenolic chemical extraction from plant samples with little input, resolving energy and environmental concerns, and enabling the ejection of biologically active compounds from plant sample during cell rupture [30,33].

Hence, this study focuses on using the "One-Factor-At-A-Time (OFAT) method" to examine the effects of UAE (ultrasonic probe) process factors, including ultrasonic frequency, extraction time, concentration of solvent, and sample/solvent ratio on extraction yield, TPC, and TFC of ethanolic extracts of *C. gileadensis* leaves. In addition, Fourier Transform Infrared Spectrometry and Gas Chromatography-Mass Spectrometry were used to assess the functional groups and phenolic constituents in the extract at the best conditions of UAE.

2. Experiments

2.1. Collection and preparation of C. gileadensis leaf

Freshly *C. gileadensis* leaves were obtained from Hadhramout situated in the geographical coordinates: 16.9304° North, 49.3653° East, Yemen. The leaves were washed in running tap-water, and dried in an air-oven for one day at 50 °C to a stable weight. The moisture content was 0.095 ± 0.03 g water/g dry sample before storage. The dried plant sample was sieved, powdered in a grinder (RETSCH - PM 100), and kept at 4 °C for later usage.

2.2. Chemicals and reagents

The reagents, and chemicals were procured from Sigma Aldrich Sdn. Bhd., Selangor, at analytical grade: ethanol (99.5 %), methanol (99.9 %), quercetin, gallic acid, sodium carbonate anhydrous (Na_2CO_3), the Folin-Ciocalteu phenol reagent, and aluminum chloride salt ($AlCl_3$). The Faculty of Chemical and Process Engineering Technology at the University of Malaysia Pahang Al-Sultan Abdullah provided the analytical laboratory where the distilled water was obtained.

2.3. C. gileadensis leaf extraction

The extraction process was carried out employing a "Qsonica Sonicators-ultrasonic probe (Q700CA Sonicator, USA)", as can be seen in Fig. 1. According to the experimental plans, 10 g of powdered *C. gileadensis* leaves samples were put within a volumetric flask before the ethanol–water solution was introduced. The following parameters were studied using the OFAT technique to examine the effects of different variables of UAE process: extraction time (5, 10, 15, 20, 25, and 30 min), ratio of solvent/sample (1:10, 1:15, 1:20, 1:25, 1:30, and 1:35 g/mL), ethanol concentration (20, 40, 60, 80, and 100 % v/v) and ultrasonic frequency (10, 20, 30, 40, and 50 kHz). When the frequency, solvent-to-sample ratio, and ethanol concentration were adjusted at 10 kHz, 10:1 mL/g, and 20 % v/v, respectively, the extraction period was varied from 5 to 30 min. After going through the Whatman qualitative filter paper, the extraction mixture was condensed to dry via a rotary evaporator that operates under vacuum condition at 50 °C "(Buchi Rotavapor R-200 paired with Buchi Vac V-500 pump, Switzerland)". The yield of extracts, TPC, and TFC for each factor was then calculated. Three experimental trials were conducted, and the average values were then calculated. For additional investigations, the *C. gileadensis* leaves extract was also chilled to 4 °C.

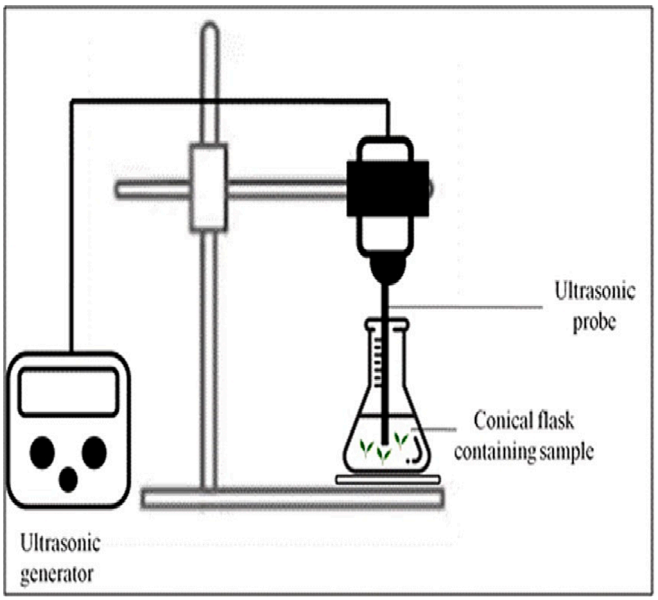

Fig. 1. Apparatus of ultrasonic-assisted extraction (ultrasonic probe) [34].

2.4. Extraction yields (Y_{EX})

The extraction yields of *C. gileadensis* leaves extracts were determined and presented in dry weight (d.w.). The yields were evaluated using Eq. (1).

$$Yield_{Extract} = \frac{Weightofextract(g)}{Weightofdriedplantpowder(g)} \times 100\% \quad (1)$$

2.5. TPC evaluation

With minor modifications, the Folin-Ciocalteu (FC) colorimetric test, which has been described before [18], was utilized to determine the total phenolic content. 10 mg of the dried extract was redissolved in 2 mL of aqueous ethanol, and 1 mL of the extract were combined with 0.1 mL of FC reagent and left at ambient temperature for 5 min. Afterwards, adding 0.5 mL of Na_2CO_3 solution, the combination was left alone for 20 min prior to its absorbance measured at 750 nm in comparison to the control (pure ethanol). via the UV–vis Spectrophotometer "[Hitachi U-1800, Japan]". Besides, the TPC concentration in the plant extract (which ranged from 50 to 500 mg/L) was estimated using the equation line $y = 0.0002x + 0.0218$, $R^2 = 0.9945$ (where: x is the calibration curve's sample concentration, and y is the absorbance at 750 nm). The TPC of the extract was calculated via Eq. (2). The tests were run three times, with the related results' mean and standard deviation (±SD) presented as "milligrams of gallic acid equivalents per gram sample dried weight [mg GAE/g d.w.]".

$$\text{TPC} = \frac{c \times V}{m} \quad (2)$$

Where;

V: extraction solvent volume (L),
m: dried sample weight used (g),
c: sample-concentration (mg/L).

2.6. TFC evaluation

With slight modifications, a previous method developed by [35] has been utilized to determine the sample's total flavonoid content. Approximately 10 mg of the dried extract was dissolved in 10 mL of ethanol to make a stock solution with a concentration of 1 g/L. An aluminium chloride solution was made by mixing 2 g of aluminium chloride with 100 mL of ethanol. The prepared aluminium chloride solution was then added to the extract in a volume of 1 mL. The absorbance of this mixture was determined at 420 nm after it has been allowed to completely react at ambient temperature for 60 min via UV–Vis. Following that, the TFC concentration in the plant extract (which ranged from 50 to 500 mg/L) was estimated using the equation line [$y = 0.0023x + 0.0374$], [$R^2 = 0.9963$] (where: x = the sample concentration from the calibration curve, and y = the absorbance at 420 nm). The TFC of the extract was evaluated through Eq. (3). The findings of the tests were presented as the mean ± SD of the corresponding results, which were performed in triplicate. The TFC was defined as "milligrams of quercetin equivalents per gram dried sample weight [mg QE/g d.w.]".

$$\text{TFC} = \frac{c \times V}{m} \quad (3)$$

Where;

V: extraction solvent volume (L),
m: dried sample weight used (g),
c: sample-concentration (mg/L).

2.7. Characterization studies

2.7.1. FTIR analysis

Fourier transform infrared spectroscopy (FTIR; Nico-let iS5 iD7 ATR; Thermo Scientific, Germany), equipped with OMNIC software, was utilized to identify the functional groups present in the leaves extract of *C. gileadensis*. Ref. [36] investigated the samples to produce IR spectra with the resolution of 4 cm^{-1} within the range of the wavelength between 500 and 4000 cm^{-1}. The sample's observed peaks were identified by comparing them to an absorption spectrum database that was predicable for the molecule's numerous bonds and groups.

2.7.2. GC–MS analysis

To identify and measure the components as described by [18], GC–MS analysis was performed on the leaves extract of *C. gileadensis*, with some changes. The extracts were examined via a TRACE GC ultra-system from Thermo Fisher Scientific, Waltham, Massachusetts, USA, equipped with a 30 m × 0.25 mm × 0.25 m Elite-5-MS capillary column. During the analysis, the column's temperature was raised from 40 °C to 220 °C at a rate of 4 °C/min. Helium gas served as the carrier gas at a flow rate of 20 mL/min, and the transfer temperature was kept at 280 °C while the injection volume of 1 L was maintained at the injector temperature of 250 °C. The MS was configured with the following parameters: EI mode, scan range of 50–600 Da, ionization voltage of 70 eV, and ion source temperature of 180 °C. Based on a library search using NIST and Wiley Registry 8″ Edition, a preliminary identification of the peaks was identified.

2.8. Statistics analysis

The Microsoft Office Excel software was used for the scientific analyses where the one-way ANOVA was employed, and the results were provided as the means ± SD of the observed values. The means of the triplicate trials were used.

3. Results and discussion

3.1. Effect of extraction time

The duration of the extraction process significantly influences the recovery of phenolic-rich extracts from plants. To ensure the preservation of bioactive compounds and achieve cost savings by minimizing process time, it is crucial to select the appropriate extraction period that allows for a thorough release of phenolic compounds from the plant material. It is significant to know that while longer extraction times generally lead to higher extract yields, there is a risk of degradation of active components. In the UAE, the effectiveness of the extraction process is determined by the interaction time between the solvent and solid plant material during the two phases of extraction. Prolonged extraction time, particularly with the aid of sonic cavitation disruption, enhances the solvent's penetration through the plant's matrix. However, there is an optimal extraction time beyond which the quality of the phenolic components found in the sample that was extracted might start to decline, so finding the right balance is essential for producing high-quality extracts [37].

Fig. 2 illustrates the extraction time's impact on the recovery yields of extract, TFC, and TPC from the extract of the leaves of *C. gileadensis*. The investigation addressed the impacts of varying extraction times (5–30 min) while maintaining a fixed ultrasonic frequency of 10 kHz, a feed/solvent ratio of 1:10 g/mL, and a 20 % v/v ethanol concentration. The results indicated that the recovery yields raised as the time of extraction progressed, reaching their peak at 15 min. At this point, the maximum recoveries were recorded as 28.9 ± 0.22 % w/w for the extract yield, 69.66 ± 2.23 mg GAE/g d.w. for TPC, and 23.22 ± 1.67 mg QE/g d.w. for TFC, respectively. However, beyond 15 min, the yields started to decline, as shown in Fig. 2. This decrease in yields might be explained by the fact that phenolic chemicals in *C. gileadensis* leaves continue to breakdown even after the 15-minute mark. Earlier research on the flavonoid components extraction from *Euonymus alatus* plant also found that the maximum yields were achieved at 15 min of extraction time [38]. These results support the research based on the extraction time. Thus, for the following stage of the "OFAT method" to evaluate the influence of the ultrasonic frequency on the extract of C. *gileadensis* leaves, 15 min (best extraction time) was chosen.

3.2. Effect of ultrasonic frequency

Acoustic conditions have a big impact on the extraction process, as highlighted in previous studies [39]. When the ultrasonic frequency increases, the formed acoustic cavitation bubble shows less intense collapse conditions. This is because cavitation bubbles require a specific time delay during the compression-rarefaction cycle to form. At high ultrasonic frequencies, the compression-rarefaction cycles become too brief, hindering the formation of cavitation bubbles. As a consequence, some cavitation bubbles require a stronger ultrasonic power to be generated [40]. In the Ultrasonic-Assisted Extraction (UAE) method, frequencies typically fall within the range of 20 to 100 kHz [41]. This power ultrasonic range is commonly used for extracting biologically active substances from biological matrices, such as polyphenols from plants and waste food items [42]. Many widely available ultrasonic probes operate at low ultrasound frequencies, typically between 20 and 40 kHz [42]. Research has demonstrated that low frequencies can produce huge cavitation bubbles in solvents used for extraction that rapidly collapse and produce microjets and strong shear forces. These conditions

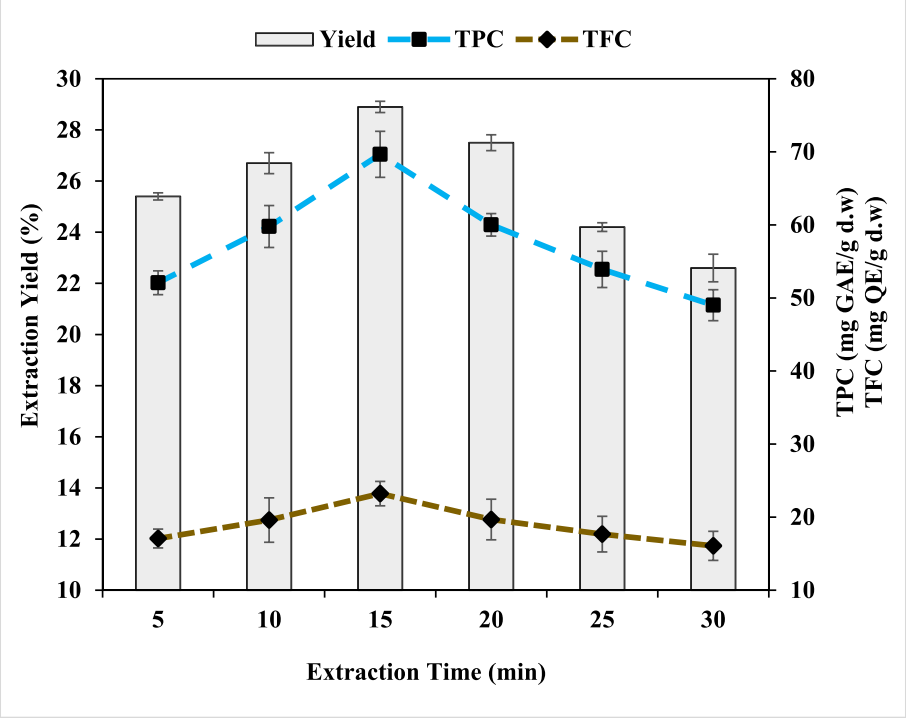

Fig. 2. Effects of extraction time (min) on the recovery yield, TPC, and TFC of *C. gileadensis* leaf using UAE.

make sure to increase the solvent penetration, enhanced cellular degradation, and higher extraction rates [43]. Consequently, when a mass transfer gradient is present, bioactive compounds are successfully recovered from plant cells. To extract plant bioactive components, researchers frequently use ultrasounds with frequencies that vary between 20 and 60 kHz [44]. This range has proven to be effective in facilitating the extraction process and obtaining desired bioactive compounds from various plant sources.

Fig. 3 depicts the correlation between ultrasonic frequency and the extraction yield, as well as the recoveries of TPC and TFC from *C. gileadensis* leaves within the frequency range of 10 to 50 kHz. During the extraction process, consistent values of a concentration of 20 % v/v ethanol, a feed/solvent ratio of 1:10 g/mL, and an extraction time of 15 min were employed. In *C. gileadensis* leaves, a noticeable increase in the ultrasonic frequency levels was observed from 10 to 20 kHz. However, as the ultrasonic frequency was further increased beyond 20 kHz, the

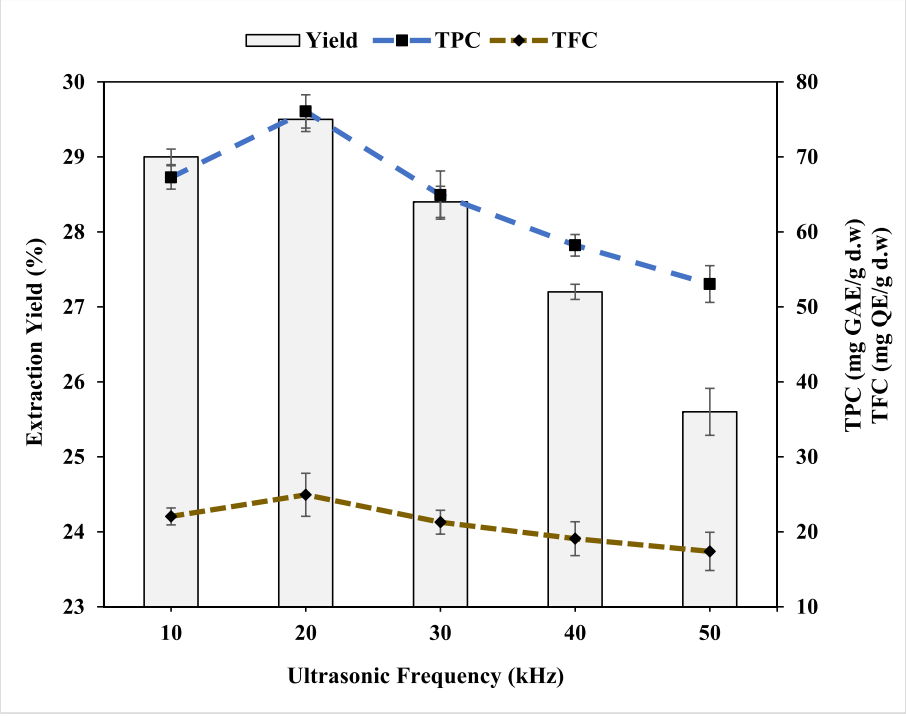

Fig. 3. Effects of ultrasonic frequency (kHz) on the recovery yield, TPC, and TFC of *C. gileadensis* leaf using UAE.

recovery yields started to decline. Fig. 3 demonstrates that the relationship between ultrasonic frequency and extraction efficiency for *C. gileadensis* leaves follows a specific pattern. There is an optimal frequency value (around 20 kHz) that leads to the highest yields of extracted compounds, while deviating from this range may result in decreased yields due to potential degradation of the bioactive compounds. Therefore, 20 kHz (the ideal ultrasonic frequency) was chosen for the next stage of the to assess the influence the ratio of the solvent/sample on the extract of C. *gileadensis* leaves. Notably, at 20 kHz, the utmost TFC, TPC, and yields of extraction yield were reported for *C. gileadensis* leaves, as shown in Fig. 3. These findings align with previous research performed by [24,45,46], who also observed that the frequency of 20 kHz resulted in the maximal yields of *Lycium barbarum L.* fruits and *Plectranthus amboinicus* leaves.

The obtained results in terms of the effect of frequency within the range 10 to 60 kHz are consistent with the expectations. On one hand, the range of frequencies responsible of an observable acoustic cavitation bubble usually starts at 20 kHz [47], which explains the increase observed initially when raising the frequency from 10 to 20 kHz, and confirms the role of acoustic cavitation bubble in the extraction process [48–50]. On the other hand, the decrease in extraction yield with the increase of frequency above 20 kHz could be attributed first to the harsher physical effects occurring at 20 kHz [51–53], namely the microjets and shockwaves the bubbles manifest at the collapse. Mechanistically speaking, the bubbles that form under lower frequency ultrasounds tend to be vaporous, of bigger volume and are characterized with relatively long acoustic cycles (longer as the frequency decreases) [54]. These characteristics make the physical phenomena associated with the collapse of acoustic cavitation bubble more violent at lower frequencies [53], and the same effects are deemed to be responsible of the observed enhancement in ultrasound assisted extraction when ultrasounds are integrated [49].

In terms of the possible sonochemical effects contributing in the ultrasound assisted extraction, references in the literature agree that though the sonochemical production of free radicals at single bubble scale is more important at lower acoustic frequencies [55], due to harsher conditions of temperature and pressure and longer reactional time [56], the number density of bubbles tend to follow the opposite trend [57]. Indeed, the number density of bubbles increase with the raise of frequency, making the overall production of radicals under low acoustic frequencies ultrasounds very limited [55]. Thus, the effects of ultrasounds in the studied process would be rather physical than chemical [49].

3.3. Effect of solvent/sample ratio

Studies have shown that a higher quantity of solvent can positively influence and enhance the mass transfer rate during extraction. However, it is essential to calculate the precise amount of solvent required when extracting bioactive compounds from plant material, as using excessive solvent may lead to increased energy demands. The solve/sample ratio has a substantial role in impacting the yield of extraction. A larger quantity of solvent creates a stronger driving force between the plant matrix and the external solvent, promoting a faster diffusion rate, which ultimately increases the extraction yields [58]. An alternative method for traditional extraction is ultrasound-assisted extraction, which offers several benefits, including reduced degradation of thermosensitive compounds, lower solvent usage, shorter extraction times, and raised yields of extraction [24,59]. To maximize recovery rates, the plant matrix must be completely submerged in the solvent during extraction. Studies by [42,60] have indicated that adding more solvent typically leads to improved extraction yields when employing traditional extraction techniques.

To recover extraction yields, TPC, and TFC from *C. gileadensis* leaves, the feed/solvent ratio was varied from 1:10 to 1:35 g/mL while keeping the extraction time at 15 min, ultrasonic frequency at 20 kHz, and ethanol concentration at 20 % v/v. Fig. 4 demonstrates that the most significant recoveries for *C. gileadensis* leaves were achieved at a feed/solvent ratio of 1:20 g/mL. When the ratio was raised from 1:10 to 1:20 g/mL, the yields of phenolic compounds raised, but they started to decline as the ratio of the feed/solvent was further increased. The probable reason for this could be the increased driving force resulting from the phenolic compounds mass transfer [61]. On the other hand, lower yields were obtained at higher solvent/material ratios, possibly due to reduced mixture density leading to faster ultrasound wave propagation. This, in turn, reduced the ultrasound power attenuation effect and increased the transfer of energy that could leads to the bioactive compounds' thermal degradation [42,62,63]. The highest yields of TPC and TFC from *C. gileadensis* leaves were achieved at a feed/solvent ratio of 1:20 g/mL, and these yields were recorded as 30.1 ± 0.21 % w/w, 88.42 ± 2.51 mg GAE/g d.w., and 28.99 ± 1.32 mg QE/g d.w., respectively. These findings align with the study conducted by [64], who investigated the solvent/solid ratio ranges of 1:5, 1:10, 1:15, 1:20, and 1:25 g/mL for obtaining the ideal yields of phenolic phytoconstituents from the *Cassia alata* plant. In their study, they discovered a total flavonoid content of 86.69 ± 1.67 mg QE/g d.w. Hence, the obtained findings were in correlation with previous results reported by [65], who found that a ratio of 1/20 g plant material/mL presented the optimal extraction efficiency for obtaining the TPC from *Melissa officinalis L.* and *Punica granatum L.*, respectively as the best extraction condition via UAE. In addition, an optimization study of bioactive compounds extracted from oregano (*Origanum vulgare*) leaves using UAE obtained that most effective F:S ratio at 1:20 g/mL to be used for extraction [66]. Overall, adjusting the feed/solvent ratio is crucial for the extraction process and obtaining the maximum yields of bioactive compounds from *C. gileadensis* leaves. So, for the assessment of the impact of the concentration of the solvent (ethanol) on the extract of *C. gileadensis* leaves, 1:20 g/mL (the best feed/solvent ratio) was chosen.

3.4. Effect of ethanol concentration

The optimal solvents' selection for Ultrasonic-Assisted Extraction (UAE) is influenced by various factors, including melting and boiling points, polarity, specific gravity, density, the solvent's affinity for the target component, and its potential effect on the activity and purity of the extracted compound [50]. Additionally, consideration should be given to how the chosen solvent interacts with the extraction parameters, intermediate, and final reaction products, as well as its reaction with the target chemicals under extraction conditions [67]. The physicochemical and bio-chemical characteristics of the solvents for extraction in UAE are crucial as they determine the properties of the extraction medium and affect the interaction between the extracted chemicals and the material being treated. Changes in extraction solvents could significantly influence the stability of polyphenols and the efficacy of therapies [42]. In the context of extracting bioactive plant components, solvents are often employed as mixture phases of organic and aqueous in varying proportions [68]. For the polyphenols chemicals extraction from plant sources using UAE, a range of organic solvents has been utilized, including ethanol, methanol, acetone, and isopropanol [42]. Ethanol has a tendency to selectively extract low molecular weight substances like non-glycosylated and glycosylated phenolic chemicals because it is a polar-protic solvent with a hydroxyl group. For instance, the peels of mango extracted via UAE, ethanol resulted in the highest total phenolic content [69]. Similarly, an ethanolic extract had the greatest total phenolic content when phenolic compounds were extracted from Thai rice cultivar bran when contrasted to other solvents like hexane and isopropanol [70]. Comparing medicinal herb *Laurus nobilis'* ethanolic extract to its water and methanol counterparts, it also revealed the greatest total phenolic content [71]. In conclusion, the selection of the solvent is essential in UAE, and factors such as solvent polarity and its effect on the extraction process should be carefully considered to obtain the desired bioactive compounds efficiently and maintain their

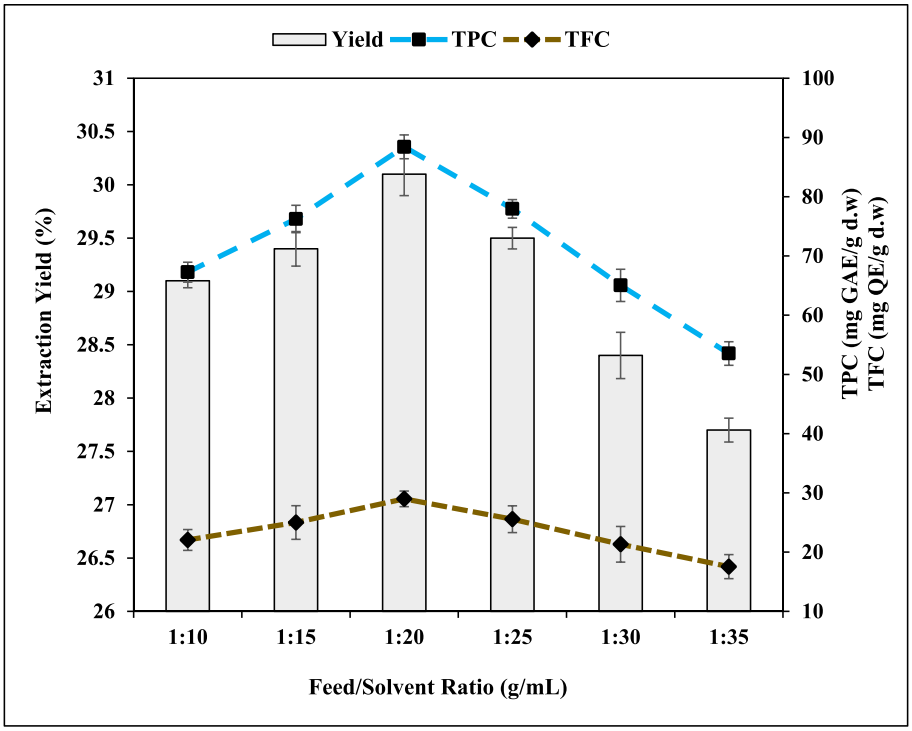

Fig. 4. Effects of feed/solvent ratio (g/mL) on the recovery yield, TPC, and TFC of *C. gileadensis* leaf using UAE.

stability and efficacy.

As represented in Fig. 5, the concentration of ethanol has a significant impact on the recovery yields of extracts, TPC, and TFC from the leaves extract of *C. gileadensis*. The study involved varying ethanol concentrations (ranging from 20 % to 100 % v/v), while maintaining a fixed feed/solvent ratio (1:20 g/mL), ultrasonic frequency (20 kHz), and extraction time (15 min).

The results demonstrated a positive relationship between the TFC, TPC, and recovery yields of extract, and ethanol concentration, up to a concentration of 40 % v/v ethanol. Beyond this point, the yields started to decline. Specifically, the highest recovery yields were observed at a water concentration of 40 % v/v ethanol. The higher affinities of phenolic chemicals for the aqueous ethanol solvent could be the cause of the rise in extraction yields after the addition of ethanol to water [34]. Changes in the polarity of ethanol brought on by the addition of water had an impact on the phenolic compounds' solubility in the solution of aquatic ethanol. The addition of water to ethanol brought about two additional effects that enhanced the mass transfer from the plant's solid matrices to the liquid-solvent. First, it increased the permeability of the plant matrix, facilitating the extraction process. Second, it disrupted the

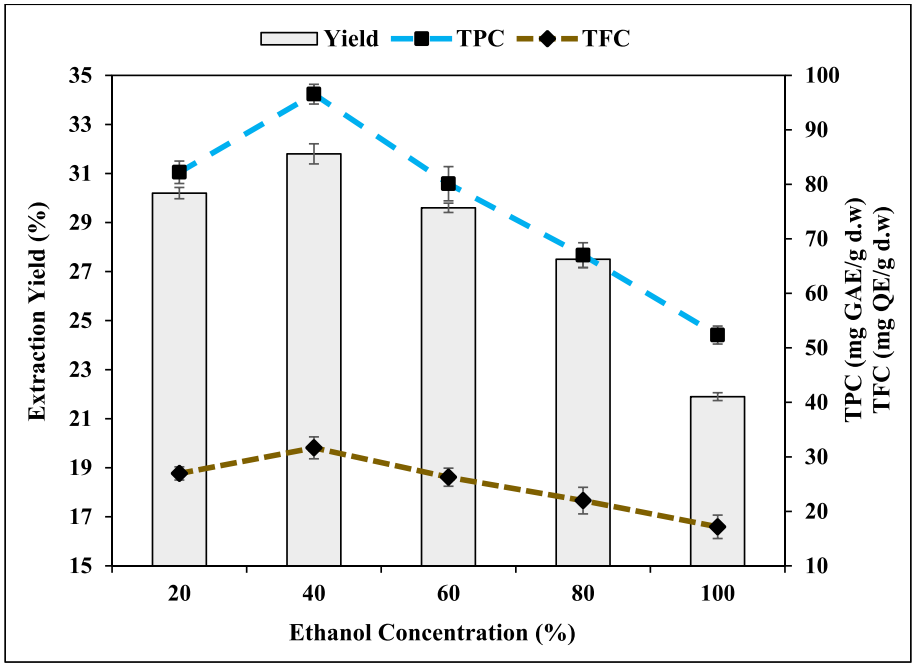

Fig. 5. Effects of ethanol concentration (%) on the recovery yield, TPC, and TFC of *C. gileadensis* leaf using UAE.

relationship between the solutes and the plant matrix, further aiding the release of phenolic compounds [72,73]. These findings align with previous research by ([74–77], who conducted extractions of phenolic components from the stems of *T. quadrispinosa* and *T. quadrispinosa Roxb* plants, achieving the highest yields under similar ethanol concentrations. More so, this result was in good correlation with the findings of [75], who studied the optimization of UAE of natural antioxidants from the flower of *Limonium sinuatum* and reported that the ethanol concentration attained the maximum extracts at 40 % v/v. In summary, the concentration of ethanol in the extracting solvent serves a significant role in determining the extraction yields of bioactive chemicals from *C. gileadensis* leaves. The ideal concentration of ethanol solvent was found to be 40 % v/v in this study, as it provided the highest recovery yields of extract, TPC, and TFC, while higher ethanol concentrations led to reduced yields. The interaction between ethanol and water affects the mass transfer and solubility of phenolic components, influencing the overall extraction efficiency.

Overall conclusion, the results obtained via UAE were about 3 times higher than the findings obtained by [18,78,79], who used to extract the phenolic compounds via solvent extraction and maceration, respectively. This reflected the advantage of obtaining higher yields using UAE in shorter extraction time and reduced solvent over the conventional method.

3.5. Characterizations

The *C. gileadensis* leaf extracts were produced at the ideal UAE conditions with extraction time of 15 min, ultrasonic frequency of 20 kHz, sample/solvent ratio of 1:20 g/mL, and concentration of ethanol of 40 % v/v; the extracts were further characterized using FTIR (for functional groups identification) and GC–MS (for tentative identification of the phytochemicals).

3.5.1. FTIR analysis

FTIR, which stands for “Fourier Transform Infrared Spectroscopy” is a technique for analysis that is utilized to find specific functional groups present in samples. In this study, FTIR analysis was performed on the extract obtained from *C. gileadensis* leaves, and Fig. 6 illustrates the outcomes. The supplementary material, specifically Table A1 (Appendix), was utilized to allocate and identify specific bands observed in the FTIR spectrum [80].

The IR spectra obtained from the extracts of *C. gileadensis* leaves were observed in the range of 4000–500 cm^{-1}. In this wavelength range, several characteristic peaks were identified, indicating the presence of specific chemical groups in the sample. A broad peak at 3335.07 cm^{-1} was observed, which is attributed to the presence of OH-groups, characteristic of phenolic compounds. Another peak at 2974.67 cm^{-1} indicated the stretching of C-H bonds. The characteristic peak at 1647.74 cm^{-1} indicated the vibration and stretching of C═C groups of protein amide, while the peak at 1381.76 cm^{-1} was indicative of the stretching of methyl (CH_2) and (CH_3) groups, it can be connected to the stretching of the carboxylic acid (C-O) of the COO-groups of carboxylates. Furthermore, sharp peaks at 1085.63 cm^{-1} and 1044.01 cm^{-1} indicated the appearance of carbohydrates/polysaccharides (C-O-C) in the sample. Overall, the IR spectra of the *C. gileadensis* leaf extract confirmed the presence of various chemical groups, as evidenced by the characteristic peaks of different functional groups in the observed wavelength range [80].

3.5.2. GC–MS analysis

The shift towards natural-based raw materials over synthetic alternatives is an ongoing effort to explore alternative options. As a result, extracts are in higher demand compared to synthetic additives, as they are commonly utilised in food and typical medical care. In this research, the chemical constituents of the extract *C. gileadensis* leaves, obtained using “Ultrasonic-Assisted Extraction (UAE)”, were identified through GC–MS analysis, and the outcomes are shown in Table 1. Overall, the analysis demonstrated that the *C. gileadensis* leaves extract contained 25 phytochemicals.

The *C. gileadensis* leaves extract was found to contain approximately 25 phytochemicals, which were tentatively identified as belonging to the groups of hydrocarbons, alcohols, esters, and fatty acids. Among these compounds, the most abundant ones include cycloheptasiloxane, cyclooctasiloxane, cyclononasiloxane, and octasiloxane, which have are claimed to possess anticancer and antimicrobial properties [81]. Additionally, the extract contains phenols with a wide-range of beneficial actions, for example; antioxidant, antiproliferative, antioangiogenic, anti-inflammatory, antimicrobial, neuroprotective, and anti-mutagenic effects [82]. Interestingly, these same phytochemicals were previously reported in the extract of *C. gileadensis* leaves, albeit at lower concentrations [18]. The variation in concentration of these phytochemicals could be assigned to the variations in the extraction methods used and the geographical location of the plant material. Overall, the *C. gileadensis* leaves extract appears to be rich in diverse bioactive compounds, which have the potential to offer various health benefits and make it a valuable natural resource for potential applications in medicine and other industries.

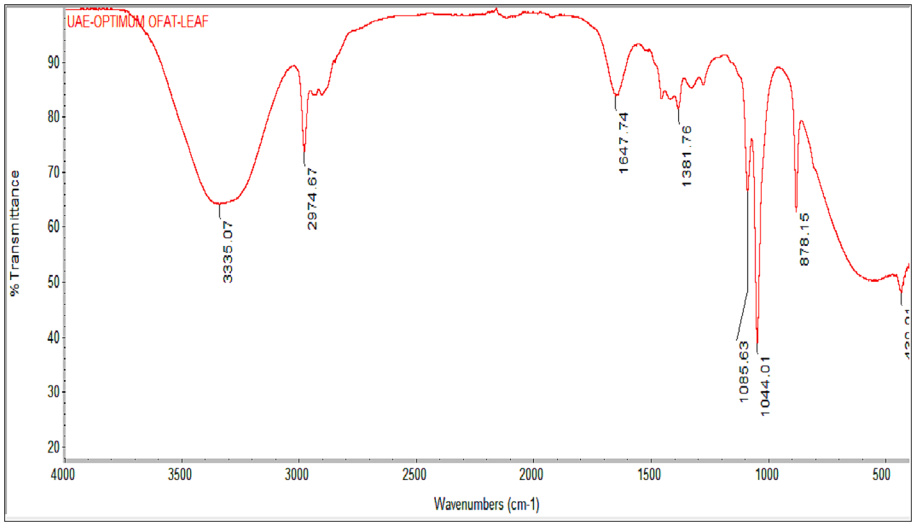

Fig. 6. IR spectra of *C. gileadensis* leaf extract from the UAE process.

Table 1
Phytochemicals of *C. gileadensis* leaves extract via GC–MS analysis.

Peak	Compound Name	Peak Area (%)	Mol. Formula	Mol. Weight	R.T (min)
1.	2,3-Pyridinedicarboxylic anhydride	8.67	$C_7H_3NO_3$	149	3.045
2.	Oxirane, 2-(1,1-dimethyethyl)-3-methyl-	3.25	$C_7H_{14}O$	114	3.115
3.	1-Butanol, 2-methyl-, (*S*)-	1.32	$C5H_{12}O$	88	3.147
4.	1-diisoprpylsilyloxycyclohexane	0.64	$C_{12}H_{26}OSi$	214	14.692
5.	Butanenitrile, 3-chloro-3-methyl-	1.91	C_5H_8C1N	117	14.778
6.	2,2,3-Trimethyl-1-phenyl-3-buten-1-one	1.45	$C_{13}H_{16}O$	188	15.487
7.	Cyclohexasiloxane, dodecamethyl-	1.01	$C_{12}H_{36}O_6Si_6$	444	18.134
8.	1-Tridecene	1.10	$C_{13}H_{26}$	182	21.029
9.	Dodecane	0.59	$C_{12}H_{26}$	170	21.305
10.	Cycloheptasiloxane, tetradecamethyl-	9.20	$C_{14}H_{42}O_7Si_7$	518	23.584
11.	Cycloprrpane, nonyl-	3.28	$C_{12}H_{24}$	168	23.711
12.	Phenol, 2,4-bis(1,1-dimethylethyl)-	7.25	$C_{14}H_{22}O$	206	24.680
13.	E-14-Hexadecenal	0.82	$C_{16}H_{30}O$	238	27.334
14.	Cyclooctasiloxane, hexadecamethyl-	11.37	$C_{16}H_{48}O_8Si_8$	592	28.514
15.	Hexandecent- 1-ol, *trans*-9-	0.63	$C_{16}H_{32}O$	240	29.825
16.	Ethanol, 2-(dodecyloxy)-	1.46	$C_{14}H_{30}O_2$	230	30.867
17.	2-Naphthalenemethanol, decahydro-.alpha.,.alpha.,4a-trimethyl-8-methylene-, [2R-(1.alpha.,4a.alpha.,8a. beta.)]-	1.10	$C_{15}H_{26}O$	222	28.993
18.	Cyclononasiloxane, octadecamethyl-	10.84	$C_{18}H_{54}O_9Si_9$	666	32.801
19.	s-Triazine, 2-amino-4-(piperidinomethyl)-4-piperidino-	0.72	$C_{14}H_{24}N_6$	276	33.368
20.	Isolongifolene, 9-hydroxy-	0.80	$C_{15}H_{24}O$	220	35.505
21.	Silane, ethylmethyl[[5-methyl-2-(1-methylethy)cyclohexyl]oxy]phenyl-	0.71	$C_{19}H_{32}OSi$	302	35.567
22.	Octasiloxane, 1,1,3,3,5,5,7,7,9,9,11,11,13,13,15,15-hexadecamethyl-	10.49	$C_{16}H_{50}O_7Si_8$	578	36.616
23.	Ethyl 14-methyl-hexadecanonate	0.80	$C_{19}H_{38}O_2$	298	38.240
24.	Heptasiloxane, hexadecamethyl-	10.02	$C_{16}H_{48}O_6Si_7$	532	40.119
25.	Cyclononasiloxane, eicosamethyl-	10.59	$C_{20}H_{60}O_{10}Si_{10}$	740	43.304

4. Conclusion

The primary goal of this study was to extract phenolic phyto-constituents from *C. gileadensis* leaves using the "Ultrasonic-Assisted Extraction (UAE)" method. The study focused on examining the yield, TPC, and TFC of the *C. gileadensis* leaves extracts obtained by systematically varying different process factors via the "OFAT (One Factor at a Time)" method. The outcomes showed that the following process settings were ideal for generating the best extraction yield, TPC, and TFC: UAE method, extraction time of 15 min, ultrasonic frequency of 20 kHz, sample/solvent ratio of 1:20 g/mL, and solvent concentration of 40 % v/v. Under these conditions, the obtained values were as follows: "yield = 31.80 ± 0.41 %, TPC = 96.55 ± 2.81 mg GAE/g dry weight, and TFC = 31.66 ± 2.01 mg QE/g dry weight". Analysis of "GC–MS" of the extract tentatively identified the existence of 25 chemical compounds, with cycloheptasiloxane, phenols, cyclooctasiloxane, cyclononasiloxane, and octasiloxane being the most abundant among them. Overall, the UAE approach demonstrated its effectiveness in extracting biologically significant phytochemicals from *C. gileadensis* leaves, making it a promising method for recovering valuable phyto-constituents from this plant material.

CRediT authorship contribution statement

Aiman A. Bin Mokaizh: Writing – review & editing, Writing – original draft, Validation, Methodology, Formal analysis, Conceptualization. **Abdurahman Hamid Nour:** Writing – review & editing, Writing – original draft, Methodology, Conceptualization. **Kaouther Kerboua:** Writing – review & editing, Writing – original draft, Validation.

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.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.ultsonch.2024.106852.

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