

Extraction and characterisation of *Cerbera odollam* seed oil as a potential plant-based corrosion inhibitor

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Abstract

Certain types of commercial corrosion inhibitors consist of a specific, tannic acid-derived compounds. Tannin compounds are antioxidants and widely found in many species of plants. In this works, oil from the seeds of the poisonous fruit, sea mango (*Cerbera odollam*), was seen as a potential candidate for a natural corrosion inhibitor. The oil was extracted via solvent extraction using a Soxhlet extractor for 6 hours at varying temperatures between 100 to 200 °C. The solvents used were hexane and methanol. Structural characterisation of active corrosion inhibition compounds in the oil samples was analysed using Fourier Transform infrared spectroscopy (FTIR) and Gas Chromatography Mass Spectroscopy (GC-MS). Thermogravimetric analysis (TGA) was done to determine the seed oil's thermal stability and decomposition temperature. The highest extraction yield was found using hexane as the solvent at 150 °C. TGA results indicated that the oil was stable at temperatures below 200 °C. The presence of carboxylic acids, as evidenced by the FTIR and GC-MS analysis, indicated possible corrosion-inhibitive properties of the extracts. Phytochemical screening tests revealed that tannin, saponin, phenol, and steroid were present in the sea mango seed oil while flavonoid was undetected.

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1.0 Introduction

The use of commercial corrosion inhibitors is an economic method to solve the world's corrosion problems. However, its high contents of chromate, phosphate, and arsenic compounds pose as a health hazard and can cause harm to the environment (Borah et al., 2020; Sulaiman et al., 2020; Genchi et al., 2022). Attention has shifted to seeking alternative and safer corrosion inhibitors from plant extracts. Several previous researches have been done to develop 'green' plant-based corrosion inhibitors (Rosli et al., 2019; Mohammed et al., 2021; Salleh et al., 2021).

Cerbera odollam, known as sea mango, is a dicotyledonous angiosperm, belonging to the family of Apocynaceae. The fruit contains active glycosides cerberin, cerberoside and odollin which are highly toxic and cause fatality if consumed (Wei et al., 2016; Menezes et al., 2018). The tree grows preferentially in coastal salt swamps and marshy areas and spreads

widely in South Asia and Southeast Asia (Chandrasekara et al., 2016; Md Isa & Suratman, 2021; Idris et al., 2022). Its fruits are available throughout the year, making it an accessible source of raw material all year round. Its young fruit is green in colour and physically looks like a mango. The fruit changes from reddish-purple to brownish-black as it gets matured. The fibrous fruit has a large seed, surrounded by a thick fibrous husk and papery outer pulp.

The seeds of the sea mango have been studied by various researchers. The fatty acid composition of the seed oil consists of oleic, followed by palmitic and linoleic. The inedible seed oil has been previously studied as an application as biopesticide and biodiesel (Kansedo et al., 2009; Ang et al., 2015; Kansedo et al., 2019). The use of non-edible oil for chemical feedstock is also an attractive option to reduce manufacturing costs.

This study focused on the extraction and characterisation of *Cerbera odollam* seed oil as a potential plant-based corrosion inhibitor. The solvent and extraction temperature were the parameters varied to investigate their effect on the oil yield. The composition of the oil was analysed using analytical equipment and phytochemical screening tests to detect the presence of active compounds such as saponin, phenol, steroid, flavonoid, and tannin that possess corrosion inhibition properties.

2.0 Methodology

2.1 Material

Reddish to brown matured *Cerbera odollam* fruits were collected around Morib Beach, (2° 44' 55.5" N 101° 26' 36.5" E) and Putrajaya Wetland (2°57'44.6"N 101°41'45.2" E), Malaysia. The fruits were cut into two and the seeds were extracted. Once the seeds were obtained from fruits, they were instantly cleaned and placed in the oven (Memmert, Germany) to prevent oxidation. The drying process was conducted for 24 hours at 80 °C with the air switch switched on to equalise the temperature and prevent decomposition. The dried seeds were then ground using a mechanical grinder to a particle size of about 850 µm.

Laboratory-grade hexane and methanol were used as the solvents to extract the seed oil. The choice of solvent was based on its polarity differences. Hexane is a non-polar solvent while methanol is a polar solvent.

2.2 Soxhlet extraction

Sea mango seed oil was extracted from the prepared ground kernels of sea mango fruits by solvent extraction using a Soxhlet extractor. Two types of solvent were tested which were hexane and methanol. About 10 g of ground seed was placed inside a paper thimble and inserted into the Soxhlet glass tube. About 200 mL of methanol was transferred into a solvent round bottom flask and placed on a heating plate. The cooling water supplied to the condenser was opened to ensure the continuous cycling of solvent. The volume of the cooling water supply was increased until all the bubbles disappear, before reducing it to an optimum volume. The extraction temperatures were set at 100, 130, 150, and 200 °C to compare the percent yield of sea mango seed oil. All samples were left for 6 hours until the samples inside the thimble changed to pale dark brown. The process was repeated in triplicate to get the average value for each condition.

The oil was then recovered using a rotary evaporator (Heidolph Instruments, Germany) where water and solvent were removed from the extract, leaving the purified crude seed oil at the bottom. The rotary evaporator was operated at 70 °C water bath temperature and at 70 rpm rotary speed. The resulting extracted oil was monitored closely until bubbles were formed, which indicated maximum residual water and solvent had been removed from the seed oil.

The extraction yield of the recovered *Cerbera odollam* seeds oil was calculated using Eq. (1).

$$\% \text{ Oil Yield} = \left(\frac{M1}{M2} \right) \times 100 \quad (1)$$

where M1 is the mass of *Cerbera odollam* crude oil extracted from the sample which has undergone excess solvent removal (g), while M2 is the initial mass of the sample before Soxhlet solvent extraction (g).

2.3 Product characterisation

The extracted sea mango oil was analysed using Fourier transform infrared spectroscopy (FTIR) (Spectrum One PerkinElmer, USA) to determine the essential functional group present in the oil at a wavelength range of 515 to 4000 cm⁻¹. Potassium bromate (KBr) was used as the background material in the analysis.

The oil was further analysed using gas chromatography-mass spectroscopy (GC-MS) (Agilent Technologies, USA) to determine its fatty acid and organic compounds composition. The gas chromatography column was equipped with a capillary column (15 m × 0.53 mm, 0.5 µm film) and Flame Ionisation Detector (FID) as the detector. Nitrogen gas was used as the carrier gas. The initial temperature of the oven was set at 120 °C for 5 minutes before raising it to 260 °C for 8 minutes at the rate of 20 °C/minute. The temperatures of the detector and injector were set at 260 °C and 230 °C, respectively.

Thermogravimetric analysis (TGA) (Mettler Toledo, Switzerland) was done to investigate its thermo-mechanical properties. The results included the determination of the extract's decomposition temperature. The test was conducted in a nitrogen atmosphere from room temperature to 800 °C with a heating rate of 10 °C/min.

2.4 Phytochemical screening

Separate phytochemical screening tests were conducted to determine the presence of active

compounds such as saponin, tannin, steroids, flavonoids, and phenolic compounds (Evans, 2002).

The saponin test (wet foam test) was conducted by placing 0.2 g of the extracted oil into a test tube containing 5 mL of distilled water. The mixture was shaken vigorously. Any frothing was taken as evidence of saponin's presence (El Aziz et al., 2019; Góral & Wojciechowski, 2020). The presence of tannins was tested by mixing 0.2 g of oil with distilled water. The mixture was stirred and filtered. Iron (II) chloride was added to the filtrate. A blue-black, green, or blue-green precipitate was taken for evidence of tannins presence (Mir et al., 2016).

Salkowski's Test was the method used to determine the presence of steroids in the oil. The oil (0.2 g) was dissolved in 2 mL of chloroform. Concentrated sulphuric acid was then carefully added to the mixture and formed a lower layer. A reddish-brown colour at the interphase is a deoxy sugar which is a characteristic of cardenolides, indicating the presence of steroids (Yadav & Agarwala, 2011).

The flavonoid test (NaOH test) was done by mixing 2 mL of 2 wt.% NaOH with 2 g of the extract to form an intense, yellow-coloured solution (Shaikh & Patil, 2020). Phenols Test was conducted by dissolving 0.2 g of the oil in an iron (II) chloride solution. A green, dirty green or dark brown precipitate indicates the presence of a phenolic compound (Soloway & Wilen, 1952).

3.0 Results and discussion

3.1 Solvent extraction of sea mango oil

The yield percentage of sea mango oil at every temperature for both hexane and methanol can be seen in Fig. 1, where it was observed that the increase in solvent temperature from 100 °C to 150 °C resulted in an increase of the yield percentage of sea mango oil. The highest oil yield was obtained at 150 °C for both hexane and methanol, which yielded 85.1% and 55.7%, respectively. This was due to the increased of solubility and diffusivity of solvent onto the samples at a higher temperature, which then improved the mass transfer. The oil yield using hexane decreased as the extraction temperature was increased to 200 °C, which could be due to the denaturation of the phytochemical compounds in the seed. The extraction using methanol at 200 °C was not conducted due to the unavailability of the sample material. However, it was predicted that extraction using methanol at 200 °C will show the same trend of decreased extraction yield. Similar phenomena have been reported by other studies which observed

the degradation of rosmarinic acid at temperatures of 150 °C or above (Hossain et al., 2011). A study reported that increasing the temperature up to 200 °C resulted in the degradation of phenolic compounds (Santos et al., 2012). It was suggested that the extraction temperature should be set near 150 °C, to effectively extract phenolic compounds from spinach leaves (Howard & Pandjaitan, 2008).

The results based on the two different solvents can provide an initial indication of the polarity of the extract. The use of hexane, a non-polar solvent, showed higher extraction yield as compared to methanol. As shown in Fig. 1, the extraction using hexane resulted in a higher oil yield at all tested temperatures. This higher yield using the non-polar solvent suggests that the seed oil consists of non-polar compounds that are more soluble in hexane than in methanol. The relative polarity and polarity index values of hexane and methanol are compared with water as shown in Table 1.

Other studies have been conducted for solvent extraction of *Cerbera odollam* using different solvents, namely hexane (Kansedo et al., 2009; Dhillon & Tan, 2016), methanol (Ahmed et al., 2008), and butanol (Mackeen et al., 2000).

Table 1: Solvent polarity values

Solvent	Relative Polarity (Reichardt & Welton, 2010)	Polarity index, P' (Snyder, 1974)
Water, H ₂ O	1.000	9.0
Methanol, CH ₄ O	0.762	6.6
Hexane, C ₆ H ₁₄	0.009	0.0

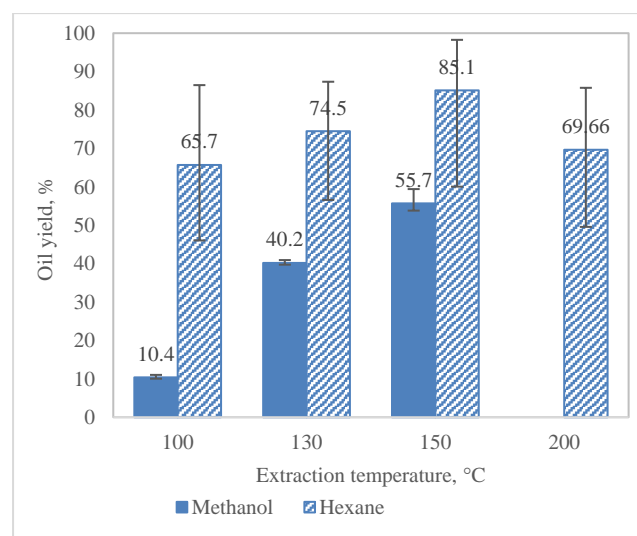


Fig. 1: Effect of temperature on oil yield using methanol as solvent

3.2 Phytochemical tests results

Phytochemical test results were summarised and presented as shown in Table 2. The tests were conducted to determine the presence of saponins, steroids, tannins, phenols, and flavonoids. Results demonstrated that the sea mango seed oil contained saponins, steroids, phenols, and tannins while the test for flavonoids showed negative results. These compounds contain oxygen and nitrogen atoms which function as the centre of adsorption. Most of the oxygen-containing constituents of the extracts are hydroxy aromatic compounds. The presence of tannins or tannic acids will form a passivating layer of ferric tannates on the metallic surface thus it will inhibit corrosion (Koerner et al., 2021; Wu et al., 2021). Saponins, which are a diverse family of natural surfactants, consist of a hydrophobic moiety which is an important aspect of an effective corrosion inhibitor to repel the aqueous corrosive species away from the metal surface. Tannins and saponins also possess antimicrobial traits that can act as antimicrobial agents and inhibit microbiologically induced corrosion (MIC) (Bismelah et al., 2019).

3.3 FTIR results

The FTIR spectrum for sea mango oil is presented in Fig. 2. The peaks at 2900 cm^{-1} and 2800 cm^{-1} represent C–H stretch and carboxylic acid O–H stretch while the characteristic peak at 1740 cm^{-1} is attributed to carboxylic acid C=O stretch. The peaks at around 1457 cm^{-1} displayed the presence of C=C aromatic rings which are indicators of tannic acid presence. Tannic acid is composed of aromatic rings with hydroxyl groups. The presence of oxygen atoms can be related to the inhibitive properties of corrosion inhibitors. These results are positive indicators that the sea mango seed oil contains compounds with potential properties of corrosion inhibition or adsorption. The hydroxyl and carboxylic groups can react with epoxide groups of coatings to form crosslinking at ambient temperature as a self-healing agent (Lee et al., 2011).

3.4 GC-MS results

The compounds that are accountable for corrosion inhibition are highlighted in the GC-MS chromatogram featured in Fig. 3. The analysis revealed some identified fatty acids in sea mango seed oil, which are composed of oleic acid ($\text{C}_{18}\text{H}_{34}\text{O}_2$ (C18:1)) and tetradecanoic acid ($\text{C}_{14}\text{H}_{28}\text{O}_2$) or myristic acid, palmitic

acid ($\text{C}_{16}\text{H}_{32}\text{O}_2$ (C16:0)), and linoleic acid ($\text{C}_{18}\text{H}_{32}\text{O}_2$ (C18:2)). Fatty acids from plant sources have been investigated and for its use as corrosion inhibition agent.

It had been previously reported that inhibition efficiency of over 92% was achieved using 1 g/L of *Cannabis sativa* L. extract that is rich in linoleic acid, oleic acid, α -linolenic acid ($\text{C}_{18}\text{H}_{30}\text{O}_2$ (C18:3)) and saturated palmitic acid (Damej et al., 2022). The AFM and XPS results showed the protection of mild steel by the adsorption of constituent phytochemicals in the leaves extract of *Crotalaria pallida* which is rich in linolenic acid, oleic acid, and linoleic acids (Jeeja Rani et al., 2021). The fatty acids formed a barrier layer on metal surfaces, thereby inhibiting the permeation of oxygen and water to the surface (Bruun et al., 2021).

3.5 TGA results

Fig. 4 represents the derivative thermogravimetric curve of sea mango seed oil. The mass of the sea mango oil starts to decline gradually from room temperature up to $100\text{ }^\circ\text{C}$. This was possibly due to the vaporisation of volatile compounds in the seed oil. The mass of oil remains stable and constant between $100\text{ }^\circ\text{C}$ to about $250\text{ }^\circ\text{C}$ which then drops rapidly, losing about 50% of the original mass. This shows that the oil is thermally stable to be used in high-temperature applications. Results from TGA confirm that the decline in the extraction yield at temperatures above $200\text{ }^\circ\text{C}$ was due to the decomposition or vaporisation of the seed oil at elevated temperature. The major degradation process corresponds to the breakdown of the chemical structures of the chemical compounds in the oil.

Table 2: Phytochemical test results

Phytochemical Tests	Observation	Presence (Yes/No)
Saponin	Formation of a stable froth	Yes
Steroid	A stable reddish-brown ring at the interface	Yes
Tannin	Green colouration appeared	Yes
Phenol	Dark brown precipitate	Yes
Flavonoid	Pale yellow solution	No

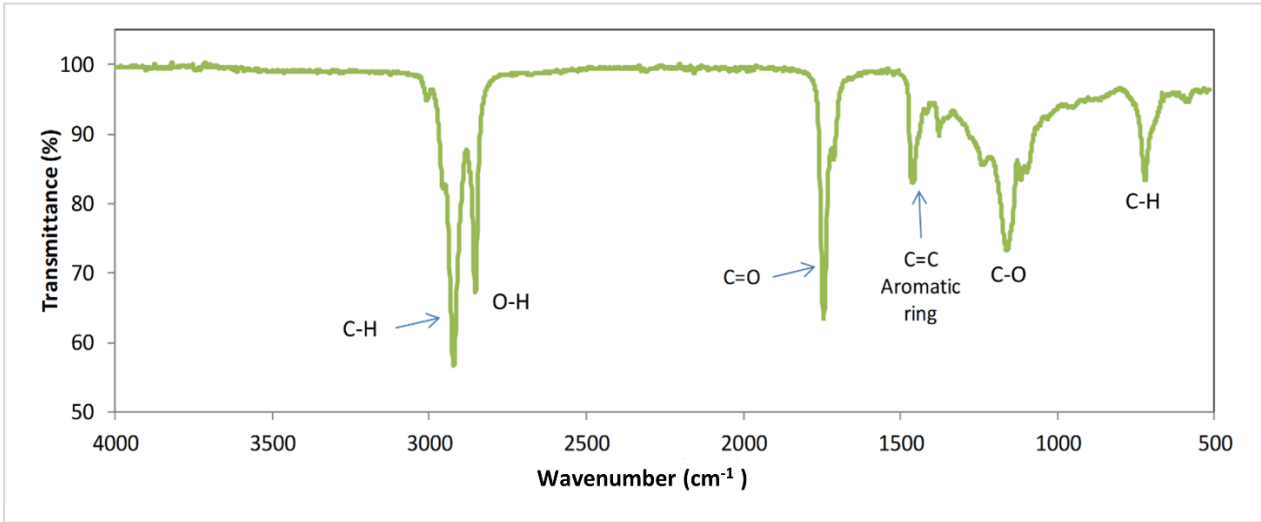


Fig. 2: FTIR spectra of sea mango seed oil

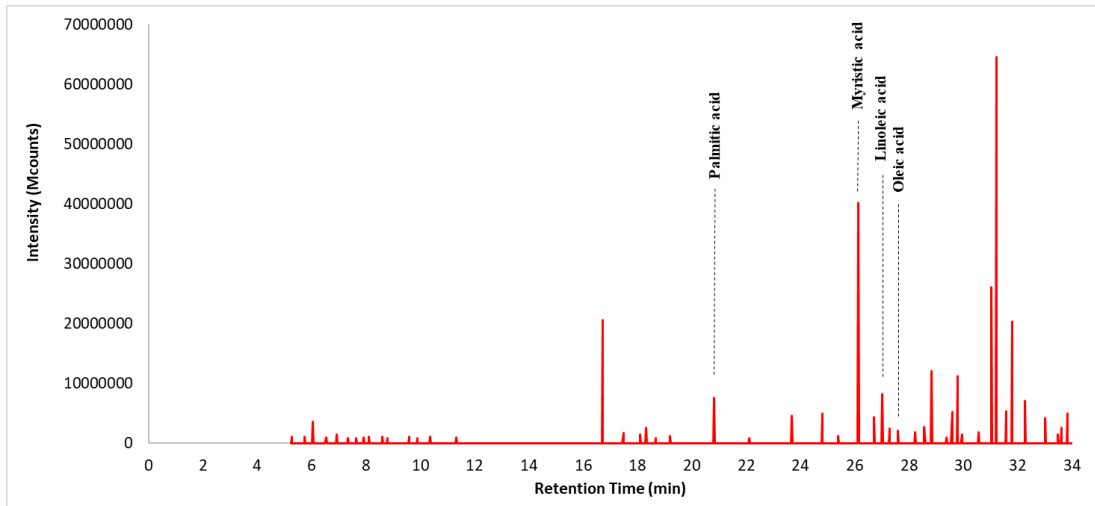


Fig. 3: Gas Chromatography Mass Spectrometry (GC-MS) of sea mango seed oil

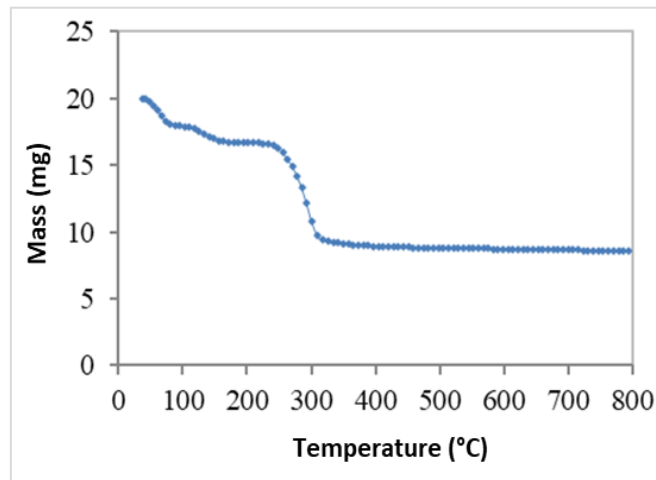


Fig. 4: TGA results of sea mango seed oil

4.0 Conclusions

In the search for more sustainable alternatives for the sake of the environment, turning to plant-based products is a positive move towards this objective. The inedible fruits of *Cerbera odollam* are available throughout the year, making them an accessible source of raw material all year round. The presence of specific chemical compounds or functional groups in the oil of the poisonous sea mango seed has the potential as a natural corrosion inhibitor to replace fossil fuel-based chemicals. These chemical compounds, such as carboxyls can adsorb on the surface of mild steel via chemisorption and prevent corrosion reactions from occurring. Saponin in the oil acts as a natural water-repellent, while tannin forms a protective layer on the metallic surface to inhibit corrosion.

Extraction of the seed oil using hexane or methanol was simple and straightforward, however further tests can be done for a more effective extraction process. Further tests can be conducted to investigate the corrosion efficiency of sea mango seed oil on metal. In conclusion, the use of plant-based, non-food sources, sustainable materials such as the sea mango deserves much attention and should be encouraged to replace fossil fuel-based chemicals.

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Contribution statement

Nor Roslina Rosli: Conceptualisation, data validation, and writing (critical review, combine, and editing original drafts); project management; supervision; **Izyan Syifaa Abdullah:** Experimental design, methodology, investigation, data analysis, and draft preparation; **Nur Adryna Mohd Rosli:** Experimental design, methodology, investigation, data analysis, and writing part of draft; **Nurul Nadiyah Zulbakeriamerudin:** Experimental design, methodology, investigation, data analysis.

Conflict of 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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