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Effect Of Ionic Liquid Pre-Treatment On Extraction Of Cinnamon Oil

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FARA serves as the technical arm of the African Union Commission (AUC) on matters concerning agricultural science, technology and innovation. FARA has provided a continental forum for stakeholders in AR4D to shape the vision and agenda for the sub-sector and to mobilise themselves to respond to key continent-wide development frameworks, notably the Comprehensive Africa Agriculture Development Programme (CAADP).
FARA's vision is to "Reduced poverty in Africa as a result of sustainable broad-based agricultural growth and improved livelihoods, particularly of smallholder and pastoral enterprises" its mission is the "Creation of broad-based improvements in agricultural productivity, competitiveness and markets by strengthening the capacity for agricultural innovation at the continental-level"; its Value Proposition is the "Strengthening Africa's capacity for innovation and transformation by visioning its strategic direction, integrating its capacities for change and creating an enabling policy environment for implementation". FARA's strategic direction is derived from and aligned to the Science Agenda for Agriculture in Africa (S3A), which is in turn designed to support the realization of the CAADP vision.

Disclaimer

Abstract

Cinnamon generally refers to the extracts from the genus Cinnamomum, which comprises over 300 species. Cinnamon is used as a spice for culinary purposes, as flavourings in cake, ice cream, etc. It is also used in making perfumes, etc. Cinnamon also has antiseptic, antimalarial, antimicrobial and antioxidant properties. The quality of cinnamon in terms of the amount of essential oil extracted and the active compounds in them depends on the region where the type of species is cultivated. Ionic liquids are compounds made up of only ions and have melting points below 100 oC. They have negligible vapour pressure and are preferred candidate solvents for environmentally friendly processes. Cinnamon bark was pre-treated using 1-butyl-3-methylimidazolium chloride ([BMIM] Cl), an ionic liquid and then distilled. Cinnamon oil yield was 1.74% as compared to 1.77% achieved using standard hydro-distillation. The pre-treated extract however contained 6 more different compounds and a higher percentage of water.

Keywords: Cinnamaldehyde, Ionic liquids, Distillation, Hydro distillation, Cinnamon oil

Introduction

Cinnamon is one of the oldest spices known to man. Historically it dates to about 2000 BC when it was used as a spice and a preservative. Cinnamon generally refers to the extracts from the genus Cinnamomum, which comprises over 300 species in the genus Lauraceae. Of the over 300 known species, C. zeylanicum (true cinnamon) and C. cassia (Chinese cinnamon) are the most common. Usually, the latter comes from China whilst the former comes from Sri Lanka. Chemically, the bark oil from these species is not significantly different, it is mainly their origin, and in the United States and the United Kingdom they are both sold under the same name, cinnamon. Sri Lanka is the major source of cinnamon, followed by Seychelles and Malagasy Republic. Indonesia, China, and Vietnam are the major suppliers of Cinnamomum cassia. (Saideswara Rao & Mary Mathew, 2012)

One of the main factors that affect the quality of cinnamon in terms of the amount of essential oil extracted and the active compounds in them is the region where the type of species is cultivated.

Cinnamon has many uses including its use as a spice for culinary purposes, as flavourings in cake, ice cream, yoghurt, pastries, and other forms of desserts. It is also used in the cosmetic and perfumery industry for making perfumes, bath oils, and other beauty products. In addition, it is also used for medicinal purposes like antiseptic, antimalarial, and antifungal agents and as insecticides. It also possesses antioxidant properties. (Singh et al., 2007)

The oil from cinnamon bark (about 1 to 4 wt%) contains cinnamaldehyde. This is the active compound in the oil and constitutes about 40 to 90% of the oil, with about 10% being eugenol (Jayatilaka et al., 1995). Figure 1 and Figure 2 show the chemical structures of these two compounds and pictures of cinnamon bark and powder, respectively.

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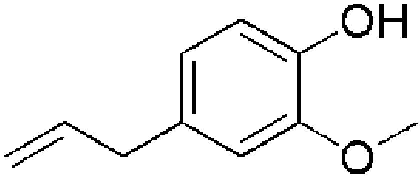

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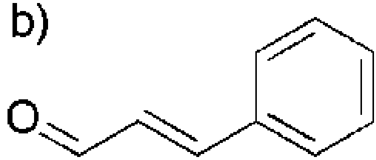

Figure 2: a) & b) Cinnamon bark sticks and c) Ground cinnamon bark.

a)

b)

c)


Figure 2: a) & b) Cinnamon bark sticks and c) Ground cinnamon bark.

The extraction of cinnamon oil from cinnamon bark involves the movement of the solvent from the external part of the solid particle of cinnamon bark to the pores to extract the cinnamaldehyde and other related compounds back into the bulk of the solvent, in other words, a process of mass transfer and diffusion.

There are various extraction methods used for the isolation of cinnamon oil or bark oil from cinnamon bark. These include steam distillation or hydro-distillation, supercritical fluid extraction, superheated water extraction, pressurised fluid extraction, and solvent extraction (Soxhlet or maceration/percolation). The analytical methods used for the quantification and separation of the compounds in the oil are, ***inter alia***, HPLC-MS, GC-MS, FTIR and TLC.

The extremely rigid structure of lignocellulosic materials which make up most biomass such as cinnamon bark has been found to dissolve in ionic liquids, newly discovered environmentally friendly solvents. Murata used ionic liquids in the extraction of Geranial, neral and geraniol from lemon grass (Murata ***et al.***, 2017). Mehta and Kumar also used an ionic liquid to dissolve Cinnamomum Cassia bark (Mehta & Kumar, 2017). Complete dissolution of biomass is believed to increase the yield of most compounds during extractions especially in conjunction with the usual less aggressive solvents (Mehta & Kumar, 2017??)

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Cassia bark (Mehta & Kumar, 2017). Complete dissolution of biomass is believed to increase the yield of most compounds during extractions especially in conjunction with the usual less aggressive solvents (Mehta & Kumar, 2017)

Ionic liquids (ILs) can be loosely defined as compounds made up of only ions but the extensive research that has been performed on these new environmentally benign liquids for the past three (3) decades defines ILs beyond that. One of the main properties of these salts that earns them the classification as ionic liquids and not molten salts is the fact that they have melting points below 100 °C unlike conventional molten salts like sodium chloride that has a melting point of 801 °C, and again one can also tentatively classify them as molten salts with melting points below 100 °C. Another difference between these two is that, ILs have organic cations instead of inorganic ones as the molten salts do. There are a number of factors that affect the melting points of these solvents but the charge, size, and distribution of charge on both the cation and the anion are the main ones. (Wasserscheid & Welton, 2008)

Another important feature that has made world headlines about these relatively new solvents is their negligible vapour pressure. In this present age everyone talks about things being made 'green' and that has initiated the quest to search for alternative solvents and processes that fall under this green category very high. Most of the conventional organic solvents being used in industry emit volatile organic compounds (VOCs), with most of them being monitored and banned due to their hazardous nature and their carcinogenic effect on the liver, lungs, and the nervous system. They also have an environmental effect on land and aquatic biomass. Ionic liquids under normal or ambient conditions are non-flammable. This makes them attractive in their use as solvents for syntheses, catalysis, and extractions. The lack of measurable vapour pressure at temperatures up to their thermal decomposition temperatures arises from the strong coulumbic interactions between the ions in the liquids. (Freemantle, 2010) They also have higher liquid ranges than normal organic solvents and due to that they are very stable at high temperatures. This makes them convenient for reactions involving high temperatures since the ILs will not degrade or vaporise at that temperature. Realistically many ILs can be decomposed at high temperatures and there is thus a limit on the maximum temperatures which can be used.

The other important feature is the fact that they can be designed to fit a particular process. This has led to them to being referred to as designer solvents and task-specific ionic liquids (TSILs) because of this unique advantage. The number of ILs that can be synthesised is enormous since the basic requirement is an organic cation and an inorganic anion. As stated earlier most of the physicochemical properties like their viscosities, densities, solubilities and miscibilities are based on the cation and anion chosen. Subsequently depending on the task, a suitable IL can be synthesised for that particular process.

The recyclability of ILs is actually possible. Some authors have proved that methods like evaporation, ionic exchange, salting (Gutowski ***et al***., 2003) and azeotropic distillation (Ressmann ***et al***., 2012) amongst others can be used to recover the IL. This particular area of ILs is still under investigation because there are other factors like energy consumption that should be taken into consideration when recycling. Another factor to also consider is the reusability of the recycled ILs.

In this work we compared the hydro-distillation of cinnamon oil from cinnamon bark and the post distillation of the cinnamon oil after the dissolution of the cinnamon bark with the IL. We also compared both the quantity of the oil extracted as well as the amount of cinnamaldehyde in the extracted oil.

Experimental Work

Materials

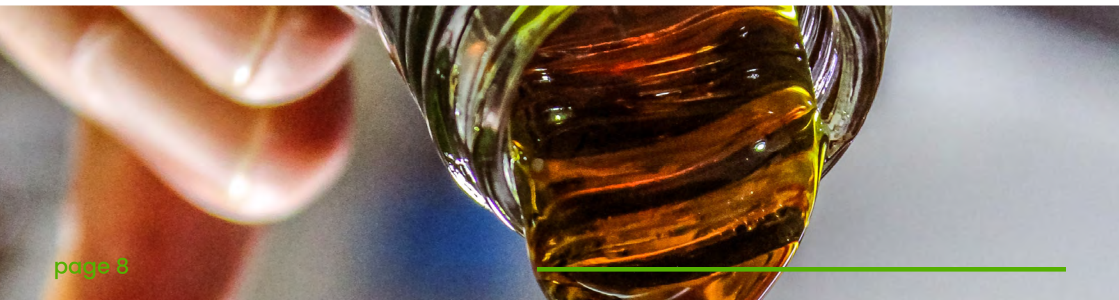
Cinnamon bark was purchased from a local Asian shop in Belfast, Northern Ireland, UK. The organic solvents used were hexane, ethyl acetate (EtOAc), and dichloromethane (DCM). These were of HPLC grade and purchased from Sigma Aldrich, UK, and used without further purification. *α*-terpeniol, anhydrous sodium sulphate, 1-methylimidazole, 1-chlorobutane, and eugenol were also purchased from Sigma-Aldrich, UK, and Cinnamaldehyde was purchased from Fluka, UK.

The Ionic Liquid, IL, (1-butyl-3-methylimidazolium chloride) [BMIM]Cl was prepared in-house, the purity and water content was checked by NMR (Carbon and Hydrogen) and Karl Fischer analysis, respectively.

Methods

Extraction of cinnamon bark oil by hydrodistillation method

The cinnamon bark was dried overnight in an oven at 100 °C prior to use and was ground using a Polymix PX-MFC 90D grinder with an outlet sieve of 500 µm attached. Approximately 5 g cinnamon bark powder was weighed and placed in an Round bottom flask (RBF) containing 150 mL water and heated at 100 °C for 8 h. Gas entry to the condenser was measured using a thermometer and maintained between 80 °C and 90 °C. The distillate was collected in a receiving flask under vacuum of 150 mbar, see Figure 4. The bark oil was extracted by washing the distillate with an equal volume of dichloromethane three times using a separating funnel. The dichloromethane phase was dehydrated over anhydrous sodium sulphate and then filtered. After solvent removal by rotary evaporation, the resulting volatile extract was stored in a freezer at -4 °C until further analysis. Figure 3 shows a setup of the distillation procedure.



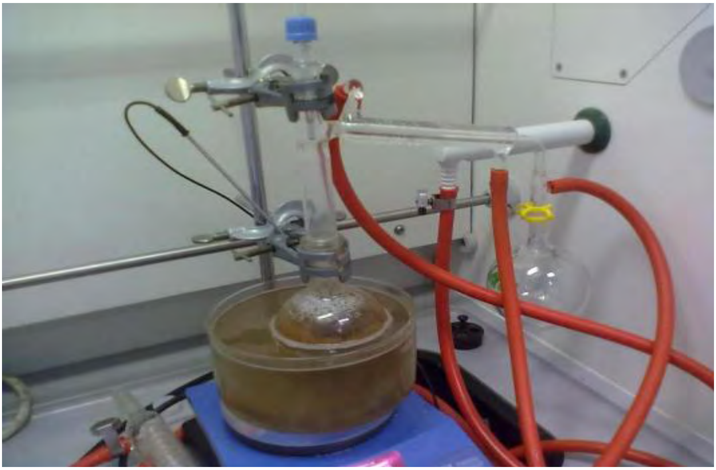


Figure 3: Distillation set up for the cinnamon bark extraction

Pretreatment with the ionic liquid

The Ionic Liquid, (IL) [BMIM]Cl previously synthesised was dried under high vacuum at 0.1 mbar, 70 °C for 3 days. The water content was 378 ppm. Apart from checking the purity of the ionic liquids synthesised, NMR was mainly used to confirm the presence or absence of ILs in the various IL dissolution extracts and also to confirm the presence of the aldehyde peaks in the extracts in the conventional methods. This technique was also used to quantify the amounts of IL in some of the IL extractions.

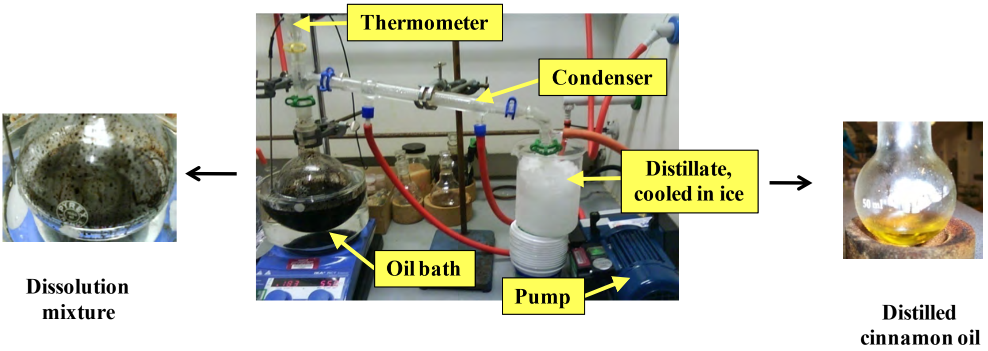
A mixture containing 20 wt% of cinnamon powder in IL was prepared and stirred for 12 h at 80 °C. The slurry formed was almost black. The distillation was then performed, with a column head temperature of approximately 40 °C. The distillate was collected under vacuum (150 mbar). The temperature of the oil bath was gradually increased from 80 to 170 °C after which 10 g of distillate was collected, equivalent to a yield of approximately 16 wt% of the cinnamon oil from the cinnamon bark. The distillate was slightly yellow in colour. The distillation set up is shown in Figure 4.

HPLC-DAD Analysis

The protocol used for this analysis was a slightly modified version of Al-Bayati ***et al***. (Al-Bayati & Mohammed, 2009) HPLC was performed using a C18 (4.6 mm i.d. x 180 mm long) Agilent column with a mobile phase of water – methanol, 40:60 (volume %). The injection volume was 20 μL and the elution was run over 30 minutes.

GC-FID and GC-MS Analysis

The GC-FID column used was a 50 m x 0.2 mm DB5 with helium as carrier gas. The temperature profile was 40 °C (1 min), 2 °C/min -> 280 °C. For the GC-MS the column used was 50 m x 0.32 m DB5 and again the carrier gas was helium. Here the temperature profile was 35 °C (1 min), 15 °C/min->50 °C (1 min), 5 °C/min -> 280 °C.



Results and Discussion

Analytical analysis of cinnamaldehyde and cinnamon oil

Standard cinnamaldehyde was used for the comparison; an HPLC chromatogram of this is shown in Figure 5.

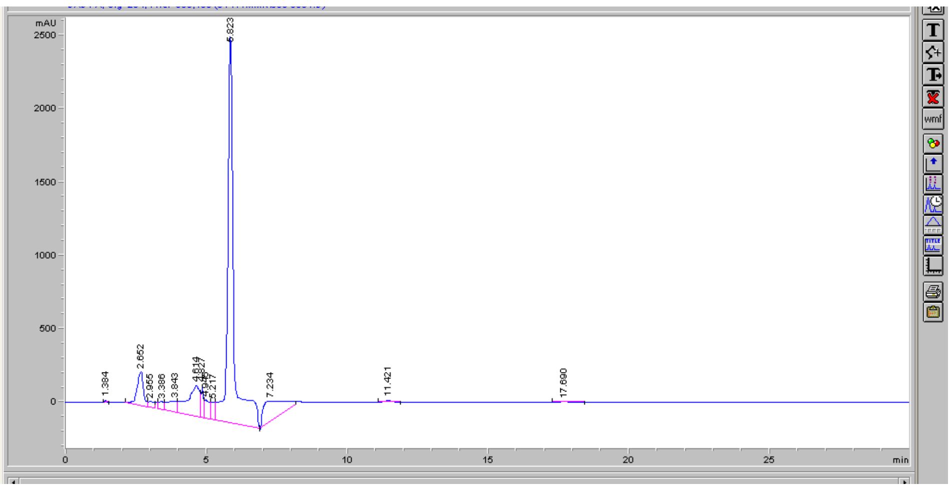


Figure 5: HPLC chromatogram of pure cinnamaldehyde

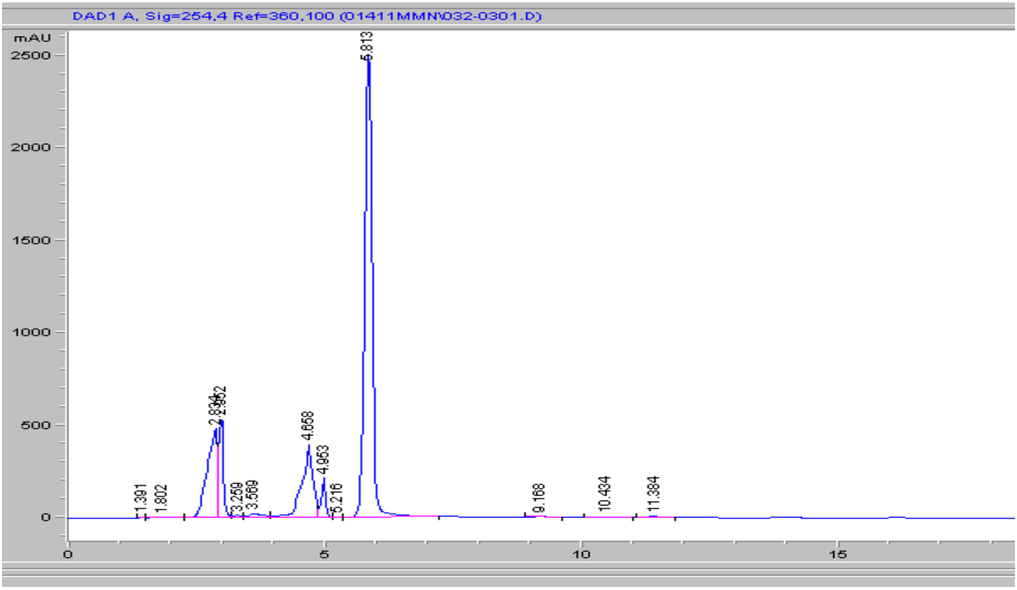


Figure 6: HPLC chromatogram of cinnamon oil from the hydro-distillation extract

All the extracts were prepared in DCM (10 mg/mL), a chromatogram of that of the hydro-distillation is shown in Figure 6. The GC-MS chromatograph of the standard Cinnamon oil, steam distillation extract and the ethyl acetate phase is shown in Figure 7. The detailed figures from the different samples are shown in Table 1.

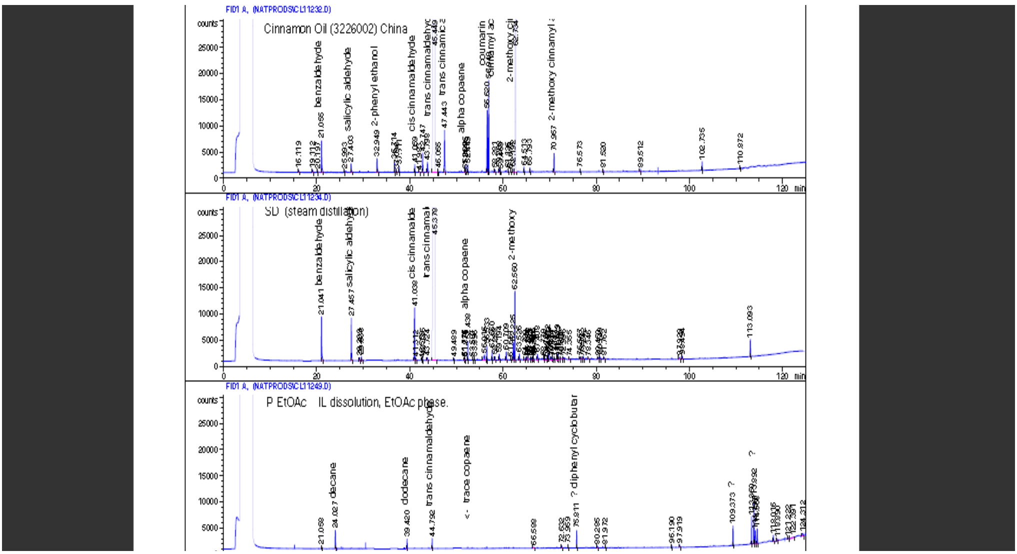


Figure 7: GC-MS chromatogram of cinnamon oil, ethyl acetate phase of the IL dissolution and the hydro-distillation extract

Table 1: A comparison of the different compounds in the different extracts using GC-MS

Sample	Meas. RT (Retention time)	Retention Index	Height	Area	Area (%)	Compound Name
Standard cinnamon oil	21.055	960	6072	41132	1.15	Benzaldehyde
	41.059	1225	1482	8896	0.25	Cis cinnamaldehyde
	45.449	1285	132887	2799301	78.34	Trans cinnamaldehyde
	47.443	1313	7990	46842	1.31	Cinnamyl alcohol
	52.449	1385	1416	6482	0.18	Alpha copaene
	56.620	1447	11749	64487	1.80	Coumarin
	56.940	1452	17562	88564	2.48	Cinnamyl acetate
Steam distillation extract	21.041	960	8378	54120	1.90	Benzaldehyde
	41.038	1224	9924	72528	2.54	Cis cinnamaldehyde
	45.378	1284	119927	2372181	83.23	Trans cinnamaldehyde
	52.438	1385	3613	16975	0.60	Alpha copaene
	56.533	1446	2698	12055	0.42	Coumarin
Hexane phase of IL extract	44.793	1276	485	2200	2.93	Trans cinnamaldehyde
Ethyl Acetate phase of IL extract	44.792	1276	1943	8638	4.25	Trans cinnamaldehyde

Effect of the various operating parameters

Composition of extracts

The composition of the extracts from the different methods used showed different levels of the various compounds as shown in Table 2.

Table 2: Various compounds and their composition (% total peak area) from the different samples analysed using HPLC

Various samples	Compound composition area (%)						
	Trans-cinnamaldehyde	Cis-cinnamaldehyde	Benzaldehyde	Cinnamyl alcohol	Coumarin	Cinnamyl acetate	α-copaene
Standard cinnamon oil	83.23	2.54	1.9	1.31	0.42	2.48	0.6
IL-distillate	69.46	1.19	0.57	-	0.23	1.15	0.23
Hydro distillation	78.34	0.25	1.15	-	1.8	-	0.18

The extract using hydrodistillation gave the highest amount of trans cinnamaldehyde while the extract from pretreatment with IL gave the highest quantity of cis cinnamaldehyde. In both cases however, the quantities of trans and cis cinnamaldehyde were below that of the purchased cinnamon oil.

Type of solvent

To produce cinnamon oil from cinnamon bark, hydro-distillation is one of the main types of extraction procedure utilised. In this case, the main solvent used is water which aids in the mild hydrolysis of the cinnamon bark and also acts as a carrier molecule for the oil being extracted. In most instances DCM is used as a post extraction solvent to isolate the cinnamon oil from the water-oil mixture.

Cinnamaldehyde being a fairly polar compound should be extracted with an equally polar solvent. In the comparison of the different solvents the difference in the weight of the bark was taken and not the mass of the oil since the former is more accurate. Table 3 illustrates the amount of material (cinnamon oil and cinnamaldehyde) extracted.

In terms of the quantity of cinnamon bark dissolved, the sample pre-treated with ionic liquid was 80.7 %, which was more than eight times that of hydro-distillation, the conventional method. This is not surprising as ILs have been proven to dissolve more than 80% of other types of cellulosic type biomass. The Hexane phase of the ionic liquid showed less cinnamaldehyde.

Table 3: Amount of cinnamon oil and cinnamaldehyde extracted from IL distillation and hydro-distillation

Extraction procedure	Bark mass change (%)	Extract yield (%)	Amount of cinnamon oil (%)	Overall yield of cinnamaldehyde (%)
IL pre-treatment distillate	80.7	74	28	1.74
Hydro distillation	5.7	3.6	46	1.77

Yield

When the bark was pre-treated with IL, almost 81% dissolved as compared to just about 6% when using the hydro-distillation method. The differences can very well be appreciated since during the concentration of the bark oil there are various losses involved. Taking for instance the distillation process, some oil could be trapped in the condenser and the distillation set up. Some authors (Ressmann ***et al***., 2012) even claim that cinnamaldehyde is slightly soluble in water, about 1 part in 700 parts of water , this could probably be why the yield was lower.

It is known that certain ionic liquids have been proven to dissolve biomass so in using the IL for the cinnamon bark extractions, the bark was broken down completely making the cinnamaldehyde easily accessible. In doing so the mass of the extract was consequently higher, as compared to hydrodistillation which is shown in Table 2.

It was observed that the IL dissolution had a higher amount of dissolved compounds (more than 8 g of the initial amount of 10 g used dissolved) taking the mass balance into consideration. This is not surprising since cinnamon bark has a wood-like structure and contains more than 60% carbohydrates (Saideswara Rao & Mary Mathew, 2012) which were soluble in the IL.

Quantity of Extract

The ^{1}H NMR of the extract from the IL pre-treatment showed a degradation of the [BMIM]Cl. Also, the extract contained more than 50% water. This water could stem from the cinnamon bark (even though it was dried in the overnight prior to the dissolution) or from the IL since the post distillation procedure took more than 12 h. It could also possibly be from the starting materials used in synthesising the IL most probably 1-methylyimidazolium or a by-product from the breakdown of the carbohydrate from the cinnamon bark by the ionic liquid.

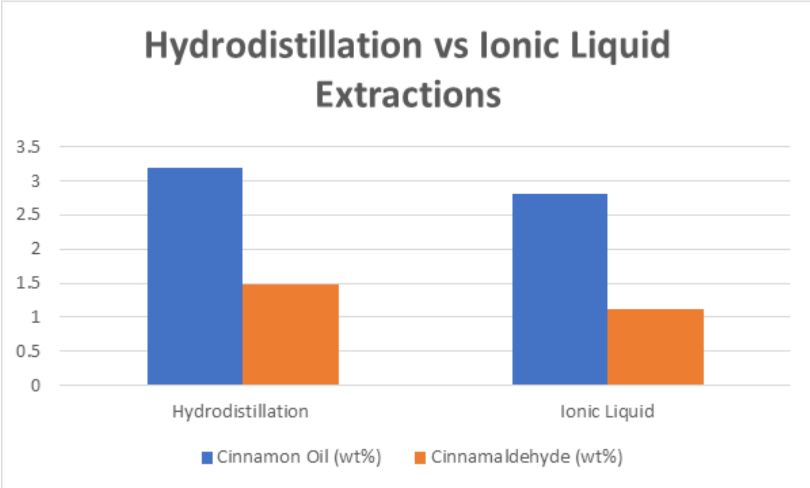


Figure 8: A comparison of the amounts of cinnamon oil and cinnamaldehyde extracted using hydrodistillation and ionic liquid.

The content of volatile oil in cinnamon bark lies normally between 1 – 6%,(Nasulhah Kasim ***et al***., 2014) and from this, depending on the origin of the cinnamon bark, it contains about 40 – 90% cinnamaldehyde. (Wong ***et al***., 2014,)(Kallel ***et al***., 2019)Cinnamomum zeylanicum Blume essential oil offers a novel approach to the chemotherapy treatment. In order to enhance its quantity/purity, the experimental conditions to produce essential oil should be more exploited. Steam distillation was used to isolate essential oil, and its conditions' optimization was carried out with the surface-response methodology. The maximum amount (2.6 g/100 g d.b. On this basis, the cinnamaldehyde content will range from 0.4 – 5.4% of the bark. From the results we had, (see table 3) it falls within this range.

Quality of Extract

The oil extracted in the hydro-distillation process is of higher quality, that is, it contains the highest amount of cinnamaldehyde, as can be seen from Figure 8. On the other hand, the yield is lower than theoretically expected figure using mass balances; apparently some of the oil might have been lost in the equipment set-up or otherwise.

The distillation process was also time-consuming; both for the hydro-distillation and the IL pre-treatment distillation. With the latter if the IL preparation and pre-treatment process is taken into consideration, it will be twice the time used for the hydro-distillation.

For the ionic liquid pre-treatment, even though it dissolved about 80% of the cinnamon bark, the percentage of cinnamaldehyde was lower (1.74%) compared to that of the hydro-distillation (see table 3)

Some of these biological compounds are confined in the core structure of the biomass and liberating them with water is often impossible. ILs are expected to break these structures down so that these compounds will be set free for them to be easily accessible by the conventional solvents. In this case, however, the IL did none of the above thereby giving a lower than expected yield of cinnamaldehyde. In fact, most of the extracts from the IL dissolutions were non-volatile since most of them couldn't be detected in the GC-MS analysis (see figure 7 and table 1).

Conclusion

The amount of oil extracted by pre-treating the bark before distillation is comparable using other methods such as hydro-distillation and also comparable to values from literature.

Considering the processes involved in using the IL in pre-treating the bark such as preparing the IL itself from other compounds and the time spent in the pre-treatment stage, it is neither economically nor environmentally beneficial to use an IL if the main aim is to extract cinnamon oil. On the other hand, if the purpose is to extract other economically beneficial compounds such as coumarin and cinnamyl acetate from cinnamon bark, then pre-treatment with ILs are essential.

Pre-treatment of the bark with IL before distillation does not give a higher yield of cinnamaldehyde though it is more efficient in aiding the extraction of other compounds from the bark. Hence hydro-distillation is a better extraction method if cinnamaldehyde is the only target compound of the extraction process.

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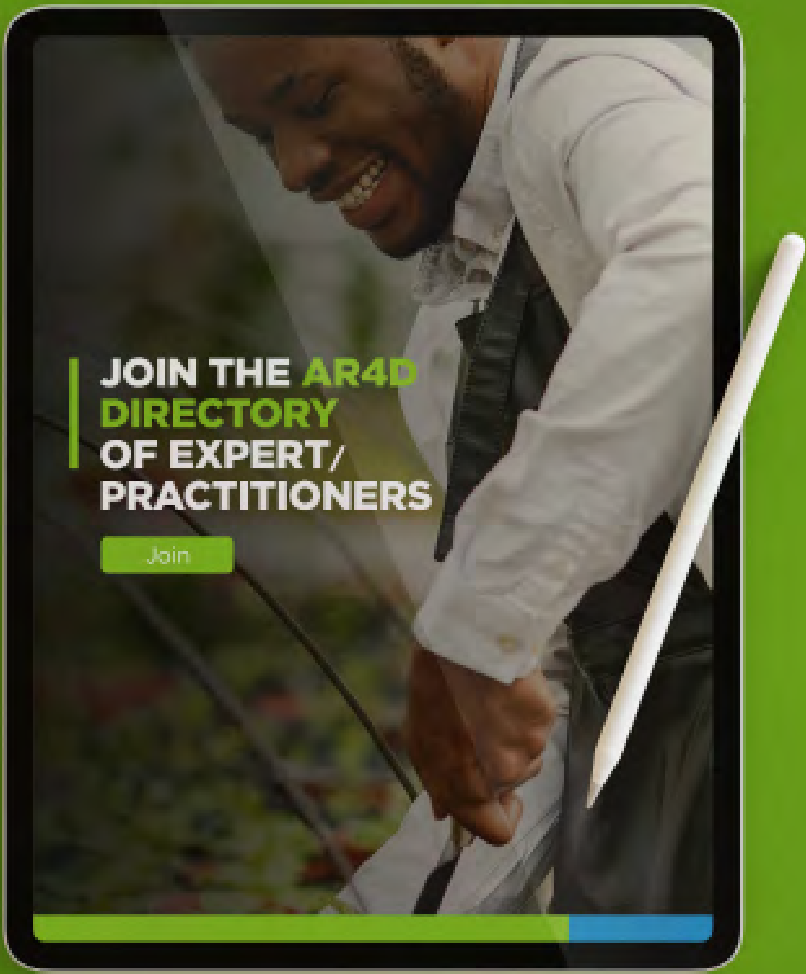

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