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Effect of Temperature on Extraction of Castor Oil from Castor Seeds Using Potential Green Solvents

Zinhle Innocentia Mkhize[1], Peterson Thokozani Ngema[1*], Suresh Ramsuroop[2*]

[1]Department of Chemical Engineering, Green Engineering Group-Chemical Thermodynamics, Steve Biko Campus, Durban University of Technology, Durban, South Africa
[2]Department of Chemical Engineering, Cape Peninsula University of Technology, Cape Town, South Africa
Email: *ngemat@dut.ac.za, *ramsuroops@cput.ac.za

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Abstract

Extraction of castor oil from castor seeds was investigated using different green solvents which include d-limonene, p-cymene, α-pinene, ethanol, and furfural at the temperature range of (323 - 413) K. The Soxhlet extraction method was employed to investigate the effect of temperature at atmospheric pressure. The focus of the study was to investigate a potential green solvent that can produce the high yields compared to the traditional solvent (hexane). The results show that at the average time of 3 hours and 30 minutes, the castor oil yield for green solvents were ranked as furfural (47.13%) > ethanol (45.37%) > p-cymene (39.15%) > d-limonene (39.13%) > α-pinene (38.11%). These castor oil yields were obtained at optimum temperatures for each green solvent. The castor oil yields were compared to the yield of hexane (31.36%) at same average time. The green solvents were recovered by using simple distillation, except furfural which was difficult to be recovered.

Keywords

Castor Oil, Yield, Soxhlet Extraction, Green Solvents

1. Introduction

The castor plant of the Euphorbiaceous family known as *Ricinus communis* has gained popularity amongst researchers because of its non-edible and renewable properties. It is a raw material in many industrial processes. *Ricinus communis* of the Euphorbiaceous family is a perineal oil crop seed that is grown in warm temperate regions [1] [2] [3] [4]. *Ricinus communis* is an old, cultivated crop of the spurge family, which represents about 0.15% of the vegetable oil produced in

the world [5] [6]. In a study conducted by [5], it was reported that castor is not a legume and that researchers should avoid the use of the term "castor bean" which is frequently found in the literature on this crop. Castor seeds contain 40% to 60% rich castor oil content by weight, which can be extracted using a suitable extraction technique [2] [7] [8]. The castor plant is believed to be native to Africa, but through cultivation, it has been distributed not only to tropical and subtropical regions but also many of the temperate countries of the globe. The castor plant can grow well at a fast rate, if situated in full sunlight and provided with ample fertilizer and water [9].

Castor plant production worldwide is above 1.3 million tons per year. India is the major producer with a production of over 60%, and China and Brazil produce 20% and 10%, respectively [10]. West Indies and some regions of Africa produce small amounts of castor beans. India largely dominates the market as 80% of castor oil is exported from there. The castor oil seed production in South Africa increased from 4000 tonnes in 1971 to 6476 tonnes in 2020 growing at an average annual rate of 1.14%, the price of castor oil was reported to have been steadily on the rise from 2002 to 2003, from a price of $946 per ton to $2390 [11]. Mubofu [7] predicted that the increase in the market would have reached $1.18 billion by the year 2020.

The castor plant varies greatly in its growth and appearance, it varies in growth habit, the colour of foliage, stems, seed size, colour, and oil content. The castor different varieties differ in resemblance from one another as shown in **Figure 1** [12]. Castor may be large perennials often developing into small trees, others behave as short-lived dwarf annuals. The tree and short-internode types are commonly referred to as giant and dwarf castor types, respectively [9] [13]. A castor plant is utilised in the production of castor oil.

Castor oil has 90% ricinoleic acid which is monounsaturated, 18-carbons fatty acid. In its structure, there is an acid group at the first carbon, a double bond between the ninth and tenth carbon followed by a hydroxyl functional group at the twelfth carbon. This multiple reaction sites properties makes castor oil and its derivatives to be found in the production of many products [14] [15] such as oil-based formulations of lubricants, grease, functional fluids and process oils, oleochemicals, reactive components for paints, coatings, inks, polymers and foams, textile finishing agents, emulsifiers, stabilizers in vinyl compounds, wetting agents, fuel and biodiesel, soap and cosmetic production, fertilizer as well as castor meal serves as cattle feed after the meal has been detoxified [16].

Castor oil is an excellent raw material in terms of price and quality, but especially this non-edible vegetable oil does not have any issues or compromise food security [17]. Recently, the use of castor oil has attracted attention for producing and optimizing biodiesel production, due to its high content of ricinoleic fatty acid and the possibility to esterify with only methanol, which assures low production costs [17] [18]. This study is focused on extracting castor oil using green solvents.

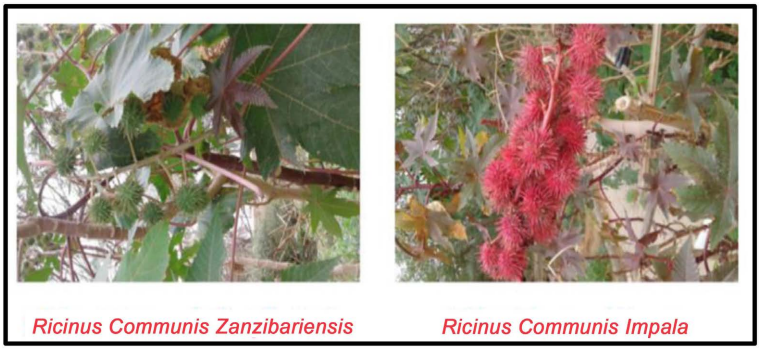


Figure 1. Different types of castor plant [12].

1.1. Green Solvents

A study by Wan *et al.* [19], reported that hexane had been used for decades to extract cotton seed oil, however regulations were put in place against hexane as it was reported to have potential health risks. Therefore, the oil industry needed to find alternative solvents to replace hexane. A study conducted by Liu and Mamidipally [20], reported on the extraction of edible oils from low oil content seeds using organic solvents. Hexane is reported to have been the solvent of choice because of its boing point range of 336 - 242 K, excellent oil-solvent properties in terms of oil solubility and ease of recovery. However, hexane as a solvent is also responsible for serious environmental problems such as fire, explosion, and air pollution, in addition to other health hazards due to its toxicity [20]. The alternative of using hexane or other petrochemical solvents is the development of sustainable methods such as using green solvents. The aim to replace petrochemical solvents used for extraction is to develop environmentally friendly processes which lead to a reduction of pollutants that adversely affect the environment [16] [21].

Some green solvents are derived from the processing of agricultural crops. These solvents are reported to be environmentally friendly solvents because they are derived from renewable resources. Kumar *et al.* [16] reported that advances on "green" approaches have great impetus in the oil industry. Several green solvents that have been derived from agricultural residues like terpenes (d-limonene, p-cymene and α-pinene), are reported to have good solubilizing properties like conventional petrochemical solvents. Terpenes are isoprene units (C_5H_8) derived chiefly from agriculture sources. D-limonene, p-cymene and α-pinene are derived from citrus peels, tree oils and pine tree respectively, and employed in many applications. In addition, to the three listed terpenes, two popular solvents derived from agricultural crops as main or byproducts through chemical or biological conversion are furfural and ethanol. These are the suite of the solvents are used in this investigation.

1.2. The Key Features of Green Solvents

There are several physicochemical properties such as vapour pressure, boiling point, aggregation or micelle concentration, biodegradability and partition coef-

ficient that can reflect environmental characteristics and the green credentials of green solvent [22] [23]. These properties can be used for preliminary assessment of their green character. For example, biodegradability or the capacity of the solvent decomposed in the environment can be measured by biochemical oxygen demand, chemical oxygen demand and total organic carbon [22]. Another example is the vapour pressure, which gives essential knowledge related to the volatile organic compounds character [22]. Green solvents are considered as safe, nontoxic and environmentally friendly [24], especially for biochemical separations in nutraceutical, pharmaceutical applications and aqueous systems because traditional petrochemical solvents are not safe [24]. The EPA [25] listed the various ways that green solvents or green chemistry can be beneficial on human health, the environment, and economic and business sector:

Human health advantage:

- The release of hazardous chemicals to air leading to damage of respiratory organs is minimized, the air is cleaner.
- Cleaner drinking and recreational water as the release of hazardous chemical waste to water is minimized.
- Increased safety for industrial workers as the risk of explosions, fires and accidents is minimized as there is less use of toxic material.
- Production is made with less waste, safer products for consumers will be produced, cleaning products and pesticides can be replaced with safer products.
- Food can be made safer as there is elimination of toxic chemicals that can enter the food chain.
- Pesticides are made to be toxic to only the pests they are engineered for and degrade after use.
- There is less exposure to toxic chemicals as endocrine disruptors.

Environmental advantage:

- Green solvents are recovered for further use or degrade to innocuous products, there is no intentional release into the environment during production or use or through disposal.
- Plants and animal encounter less harm from toxic chemicals release into the environment.
- The potential of global warming, ozone depletion and smog formation are lowered.
- There are minimized chemical disruptions of the ecosystem.
- The use of hazardous waste landfills is minimized.

Business and economic advantage:

- Chemical reactions give off higher yields, while consuming smaller amounts of feedstock to obtain the same amount of product.
- Synthetic processes are minimized, allowing for faster production of products.
- Increased plant capacity
- Saving on energy and water

- Reduction of waste, elimination of costly remediation, hazardous waste disposal, and end of pipe treatments
- Replacement of purchased feedstock by a waste product.
- Less product is required to achieve same function through better performance.
- Reduction of petroleum products, slowing their depletion and avoiding their hazards and price fluctuations
- Improved competitiveness of chemical manufactures and customers
- Increased consumer sales from safer product labelling and reduced footprint through increased throughput

1.3. The Extraction of Castor Oil

The extraction of castor oil can be done through a series of process either through mechanically pressing (hot or cold) the castor seeds or by solvent extraction. It can also be done using the combination of the mentioned process [26]. There are different types of extraction processes, which include traditional method, solvent extraction, conventional extraction and Soxhlet extraction. This investigation is uses the Soxhlet extraction process to extract castor oil using green solvents.

1.4. Soxhlet Extraction

A Soxhlet extractor is a piece of laboratory apparatus invented in 1879 by Franz von Soxhlet. It was originally designed for the extraction of a lipid from a solid material [27]. The Soxhlet extraction method is typically used when the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. It allows for unmonitored and unmanaged operation while efficiently recycling a small amount of solvent to dissolve a larger amount of material. The description of the Soxhlet extraction apparatus is presented in more detail in section 2.2. **Table 1** listed an advantages and disadvantages of Soxhlet extractor [28] [29].

In this study, the effect of temperature on the yield of castor oil using potential green solvents were investigated. The selected green solvents: d-limonene; α-pinene; p-cymene; furfural and ethanol and the investigated temperature ranges are presented in **Table 2**. The experiments were conducted at atmospheric pressure. The castor oil yield was calculated using the weights or mass of crushed castor seeds before and after extraction. The separation of solvent-oil mixture was carrier out with a simple distillation, where solvent and castor oil were recovered, respectively. Further, the refractive index and density for the recovered solvent and castor oil produced were measured to determine the stream purities and to check if any has reaction taken place during extraction.

2. Experimental

2.1. Materials

Before using the green solvents, the DMA 4100 M refractometer from Anton Paar which has an accuracy of ± 0.0002 nD was used to measure the density and

Table 1. Advantages and disadvantages of Soxhlet extractor [28] [29].

Advantages	Disadvantages
The sample is repeatedly brought into contact with fresh portions of extractant, which facilitates displacement of the transfer equilibrium.	Samples are usually extracted at the solvent boiling point over long periods, which can result in thermal decomposition of thermolabile target species
The system remains at a relatively high temperature by effect of the heat applied to the distillation flask reaching the extraction cavity to some extent.	Large amount of extractant wasted which is not only expensive to dispose of, but also the source of additional environmental problems.
No filtration is required after leaching	Long time required for extraction
Low cost of the basic equipment	Conventional Soxhlet device provides no agitation
Soxhlet extraction is a very simple methodology that requires little training	The Soxhlet technique is limited by extractant and difficult to automate
Sample throughput can be increased by performing several simultaneous extractions in parallel	Energy consuming and conventionally used high amount of petroleum solvents
Extract of more sample mass than most of the latest alternatives (microwave-assisted extraction, supercritical fluid extraction, etc.)	The large amounts of extractant used call for an evaporation concentration step after extraction.

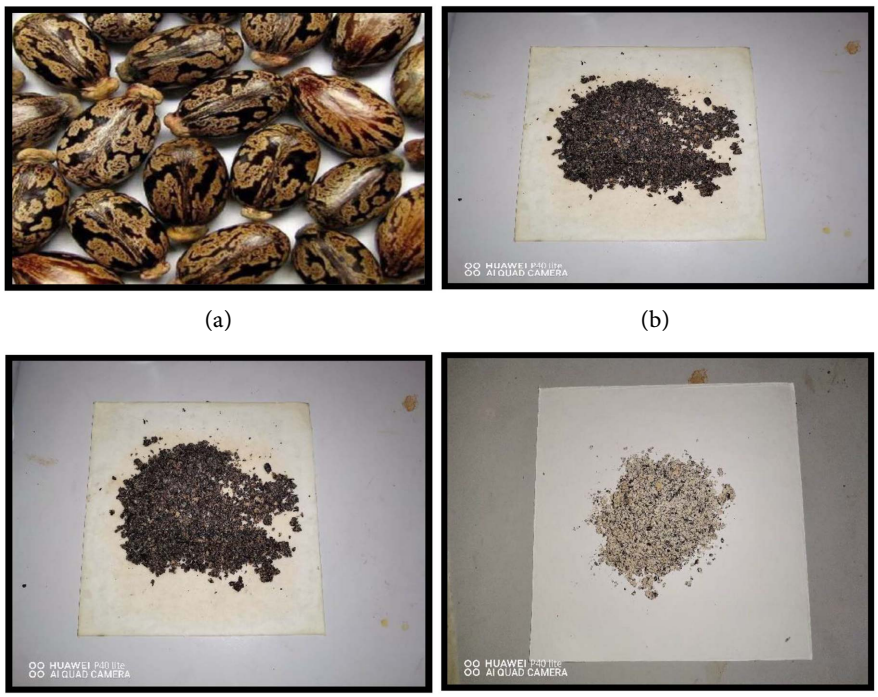
Table 2. Temperature ranges investigated for the potential green solvents.

Solvent	No. Points	Temperature (K)
d-Limonene	7	353.15 to 403.15
Ethanol	5	323.15 to 263.15
p-Cymene	5	353.15 to 393.15
α-Pinene	5	348.15 to 413.15
Furfural	6	346.15 to 383.15

refractive index. The measurements were used to check the purities of potential green solvents. Table 3 presents detailed information on the supplier and purities of the green solvents including hexane used in this investigation. Castor seeds were cleaned of debris and stones. Castor seeds were crashed using the mortar and pestle to deshell. The seeds were first deshelled by pressing the seeds until the covering popped open exposing the soft inside of the seeds as shown in Figures 2(a)-(d). The crushed deshelled seeds were then dried in an oven at 348.15 K for 18 hours to remove moisture. The size distribution was not taken into consideration because it was not an objective of this study. After 18 hours the crushed castor seeds were further air-dried for 48 hours at a temperature of

Table 3. The green solvents and traditional solvent information used in this study.

Solvent	Chemical formula	Molecular weight (g/mol)	CAS number	Purity (%)	Boiling points (˚C)	Supplier
d-Limonene	$C_{10}H_{16}$	136.24	138-86-3	97	176.00	Merck
Ethanol	C_2H_5OH	46.07	64-17-5	99	78.37	Merck
p-Cymene	$C_{10}H_{14}$	134.21	99-87-6	99	177.00	Merck
α-Pinene	$C_{10}H_{16}$	134.21	7785-26-4	98	156.00	Merck
Furfural	C_4H_3OCHO	96.10	98-01-1	99	161.60	Merck
Hexane	C_6H_{14}	86.81	110-54-3	99	69.10	Merck



(a) (b)

(c) (d)

Figure 2. (a) The castor seed before being processed; (b) Crushed castor seeds before extraction and drying; (c) Crushed castor seed after drying before extraction; (d) Crushed castor seed after solvent extraction and drying.

293.15 K before using them. An analytical mass balance (Ohaus Adventurer balance, model No. AV 114) which was calibrated by the supplier with the stated manufacturing uncertainty of ±0.0001 g in mass was used to measure the weight before and after the experiment.

2.2. Apparatus

Figure 3 shows the schematic diagram of the experimental set-up used in this study. **Figure 4** shows the Soxhlet apparatus used in this study [30]. The Soxhlet extractor is used when the desired compound has limited solubility in a solvent.

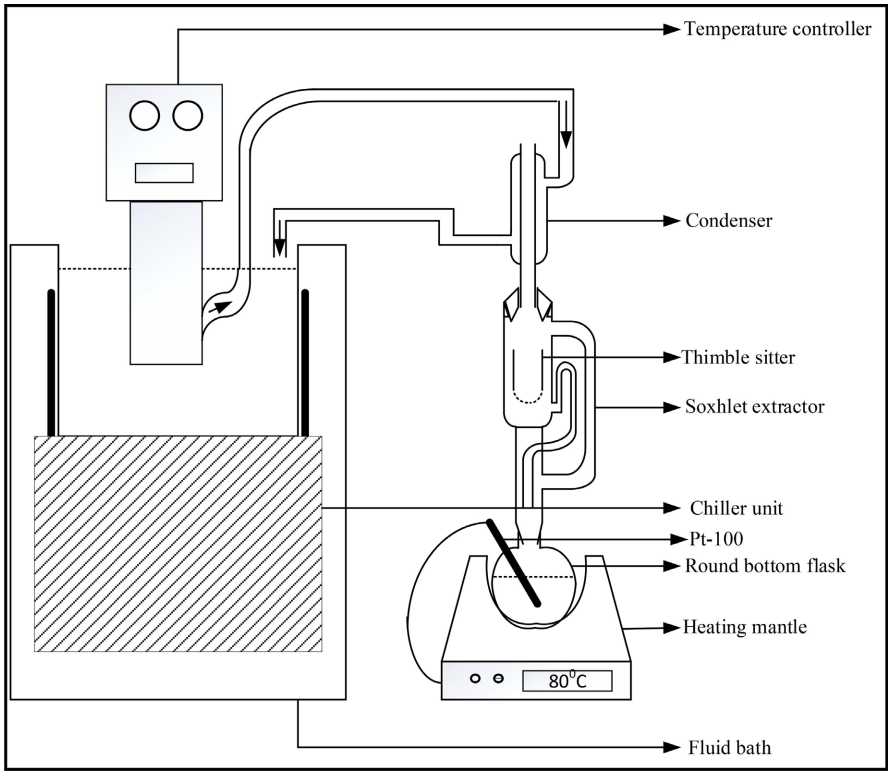


Figure 3. Schematic diagram for experimental set-up.

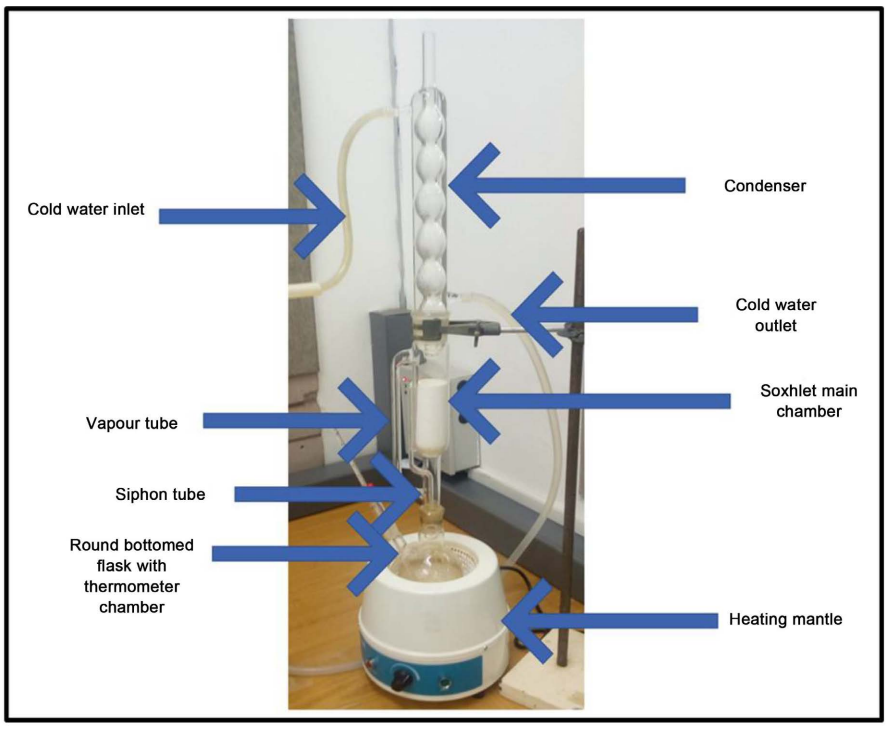


Figure 4. An assembled Soxhlet extractor.

It consists of three main sections: a percolator (boiler and reflux) which circulates the solvent, a thimble (usually made of thick filter paper) which retains the

solid to be extracted, and a siphon mechanism that periodically empties the thimble as shown in **Figure 4**. The Soxhlet extractor consists of the condenser; siphon tube; Soxhlet extraction main chamber; vapour tube; cooling fluid outlet; cooling fluid inlet and a heating mantle. The digital temperature control ISOLAB heating mantle CE is fitted with a ceramic Pt100 with manufacturing uncertainty of ±0.3 K. The heating mantle was used as the heat source to boil the solvent. The vapour tubes provide a path for the solvent vapours to flow up to the main chamber. The siphon tubes provide a path for the extracted fluid to flow down to the round-bottomed flask [30].

The condenser plays an essential role in condensing the solvent vapours that flow down as droplets into the thimble. The condenser consists of the inlet and outlet for the cooling fluid. The cooling fluid ethylene glycol at a supply temperature of 268.15 K flows counter current with solvent vapours. The cooling fluid was supplied at the temperature of 268.15 K to ensure all solvent vapours were condensed.

The viscosity of the produced castor oil was measured using the Anton Paar oscillation U-tube viscometer DSA 5000 M at 298.15 K, and the DMA 4100 M refractometer from Anton Paar was used to measure the density and refractive index of castor oil as well as the solvents used.

2.3. Experimental Method

The round bottom flask, Soxhlet apparatus and condenser were flashed with acetone to remove impurities. An oven at the temperature of 313.15 K was used to dry or remove acetone residual in all items before using them. The clean 100 g deshelled castor seeds were weighed for initial mass before crushing. The seeds were placed in a ceramic mortar and crushed using a ceramic pestle. The crushed seeds were placed in a conventional heating oven for drying at the temperature of 333.15 K. The sample of 15 g of dry crushed seeds was placed in the thimble, and the thimble was then placed into the main Soxhlet chamber [30]. The sample of 180 ml of solvent was poured into the round bottom flask that was placed into the heating mantle. The Soxhlet extractor was connected to the round bottom flask. The initial temperature of 323.15 K was set to the heating mantle. The extraction process was carried out for 3 hours and 30 minutes. The process was stopped. The thimble was lifted using a tweezer and allowed to drip dry. The wet castor seeds were emptied onto filter paper and placed in the oven for drying for 60 minutes at a temperature of 348.15 K. After drying the seeds, the final mass after extraction was recorded. The process was repeated for all the solvents at different temperatures as shown in **Table 1**. The extraction was carried out using d-limonene; α-pinene; p-cymene; furfural and ethanol as solvents [30].

2.4. Castor Oil Recovery

The amount of castor oil produced to the amount of dry castor seeds that were used can be defined as the yield, the yield is calculated by calculating the weight of dry castor seeds before the extraction and comparing the weight of castor

seeds after extraction and drying has taken place. The yield (Y) was calculated using Equation (1) [30] [31]

$$\%\text{Yield} = \frac{y_1 - y_2}{y_1}(100) \tag{1}$$

where y_1 is the weight of the castor seeds before extraction (kg), and y_2 is the weight of the seeds weight (kg) after extraction.

2.5. The Recovering of Solvent and Oil

The process of separating the solvent-oil mixture collected in the round bottom flask was carried out using a simple distillation unit. It consists of a heating mantle, thermometer, condenser, and elbow used for directing flow to the beaker. The temperature was set closer to the boiling point of the solvent used. The vapours of the solvent were condensed and collected in the beaker. The simple distillation process was stopped when no further droplets of solvents were produced. The liquid remaining in the still was a pale-yellow extracted castor oil. The recovered oil and solvent were then analysed to determine purity and relevant properties.

3. Results and Discussion

The green solvents were checked for purity to ensure that there is no contaminant. The test system for hexane was conducted to validate the procedure and reproducibility of the existing data of hexane measured by [31]. The absolute average deviation was calculated in Equation (2) and the results are presented in Table 4.

$$AAD = \left| \frac{P_{lit} - P_{meas}}{P_{lit}} \right| \tag{2}$$

The effect of temperature in the production of castor oil using potential green solvents, which includes ethanol, furfural, d-limonene, p-cymene and α-pinene is presented in Figures 5-9.

The general observations with all the solvents with the exception of α-pinene showed an increase in extraction yields with an increase in solvent temperatures. The oil solubility in solvent increases with extraction temperature, high temperature also has a positive effect on viscosity and diffusivity of oil as viscosity decreases resulting in greater diffusion rates of the oil in the matrix as the extraction temperature increases. The higher temperatures also resulted higher solvent flowrates through the thimble resulting in higher extraction rates.

Figure 5 shows that for ethanol, an increase in temperature led to an increase in efficiency of mass transfer and solubility of solvent-solute hence extraction there is an increase with temperature until exceeding the boiling point of the solvent. At higher temperature of 90˚C, which is above the boiling point therefore there is not enough contact between the castors crushed seeds and solvent. This leads to lower castor oil yield of 28.36%.

Table 4. Castor oil yield obtained using hexane.

Temperature (K)	Time (hours)	Literature (%) [31]	Measured (%)	AAD
329.15	2	31.99	31.36	0.019
333.15	3	33.44	33.67	0.007

$$AAD = \left| \frac{P_{lit} - P_{meas}}{P_{lit}} \right|.$$

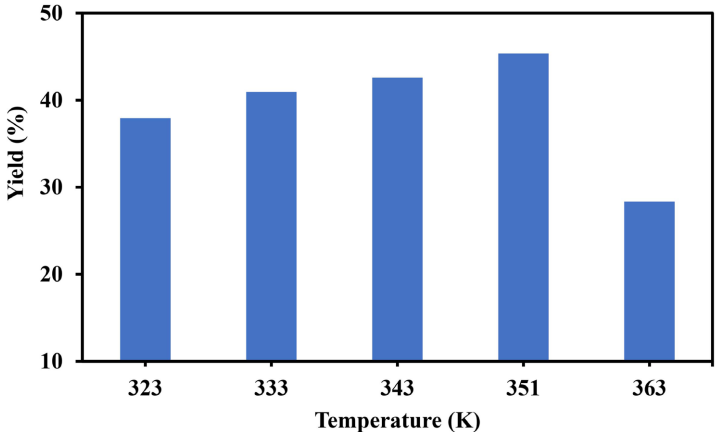


Figure 5. Effect of temperature on castor oil yield using ethanol as solvent.

Figures 6-8 revealed that the yield of castor oil obtained from the amount of vapours formed that increase with temperature which leads to increase solvent flowrate into the thimble hence greater extraction. The circulation of vapours shows an efficiency of mass transfer. The general trends also showed a decrease in extraction rates at solvent temperatures greater than 100˚C. This could be attributed to gumming of the oil in the matrix hence leading to lower yields. Figure 9 shows the castor oil yield decreases as the temperature increases. This could be attributed to the complex structure of *α*-pinene.

All investigate green solvents were showing higher yields than hexane. The results obtained on the average time of 3 hours and 30 minutes, the castor oil yield for green solvents were ranked as furfural (47.13%) > ethanol (45.37%) > *p*-cymene (39.15%) > *d*-limonene (39.13%) > *α*-pinene (38.11%). These castor oil yields were obtained at optimum temperatures for each green solvent. The castor oil yields were compared to the yield of hexane (31.36%). It is recommended that hexane can be replaced by ethanol although furfural has a higher yield. The difficult recovery of furfural indicate the possible presence of azeotrope formation in oil-furfural system. Hexane has the potential for causing serious environmental problems such as fire, explosion, and air pollution, in addition to other health hazards due to its toxicity [20]. The use of green solvents is recommended as they produced higher castor oil yield than hexane. This result could be explained to be attributed to the difference in solvent polarities of the different solvents. Terpenes and alcohols are generally known to be more polar

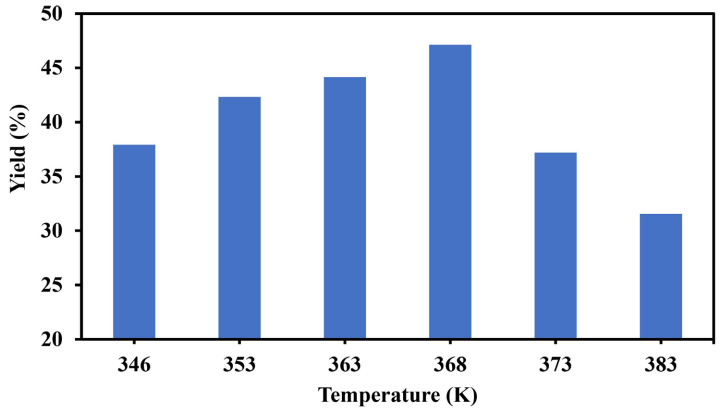


Figure 6. Effect of temperature on castor oil yield using furfural as solvent.

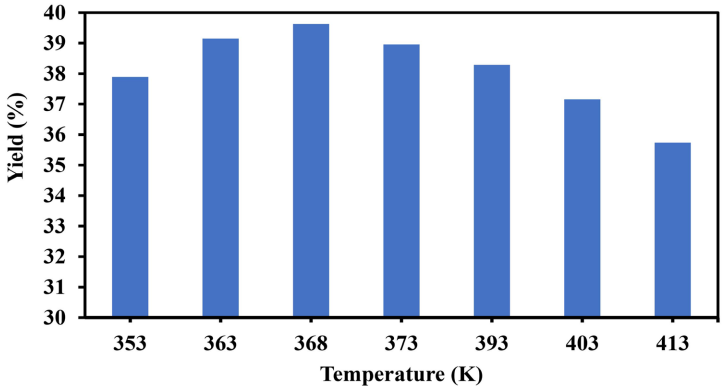


Figure 7. Effect of temperature on castor oil yield using d-limonene as solvent.

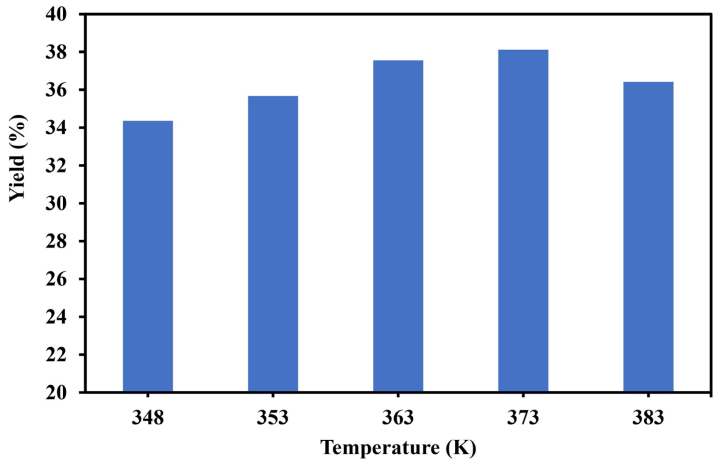


Figure 8. Effect of temperature on castor oil yield using *p*-cymene as solvent.

than hexane. Green solvents are environment-friendly, not harmful to humans, reduction of pollutants and replace petrochemical solvents [16] [21].

It was found that furfural was difficult to recover compared to other green solvents. The green solvents were recovered by using simple distillation, except furfural which was difficult to recover. Consequently, ethanol is the best green solvent can be utilized in chemical separation processes industries. Ethanol has

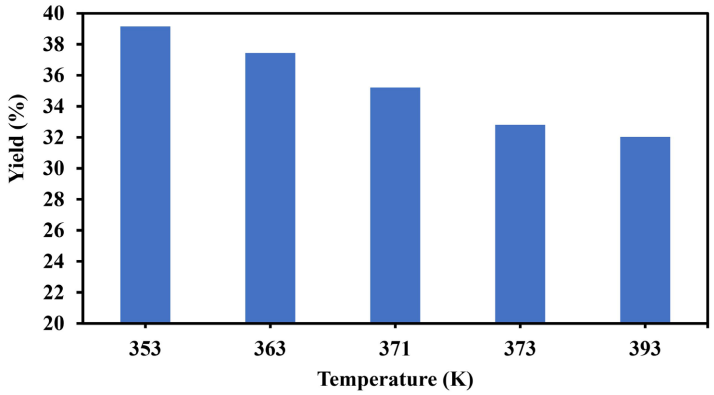


Figure 9. Effect of temperature on castor oil yield using *α*-pinene as solvent.

Table 5. Physical properties of the recovered green solvent.

Solvent	This study Refractive index (nD)	Literature Refractive index (nD)	Absolute Error	This study density (kg/m³)	Literature density (kg/m³)	Absolute Error
Ethanol	1.3661	[a]1.3614	0.0047	789.22	[a]789.47	0.25
d-Limonene	1.4873	[b]1.4701	0.0185	840.61	[d]838.35	2.26
p-Cymene	1.4886	[c]1.4912	0.0026	861.88	[c]857.38	4.50
α-Pinene	1.4706	[c]1.4631	0.0075	860.83	[c]858.10	2.73
Furfural	–	[b]1.5235	–	–	[b]1159.92	–

[a][34]; [b][35]; [c][36]; [d][37]; Absolute error $= \left|P_{lit} - P_{meas}\right|$.

Table 6. Physical properties of castor oil produced.

Parameter	Literature	This study	Units	Absolute Error
Refractive index	[a]1.4764	1.4761	nD	0.0003
Density	[b]958.00	958.12	kg/m³	0.12
Viscosity	[a]9.307	9.390	Pa.s	0.08

[a][37] [38]; [b][39]; Absolute error $= \left|P_{lit} - P_{meas}\right|$.

good advantages over petroleum solvents or traditional solvents. Ethanol is not expensive, not harmful and cost effective. This corresponds to economical utilization of agricultural residues for income generation, reduced load on waste management system, stable burning resulting into lesser generation of pollutants and air pollution [32]. In addition, it decreases the environmental pollution by rapid degradation, superior quality products as the residual solvent in the product is lesser harmful to the consumer as compared traditional solvents [32]. The refractive index and the density were measured for the recovered solvents compared to the literature values as presented in Table 5. This was done to ensure no trace of castor oil in the recovered solvent or any unexpected reaction. The absolute error was calculated using Equation (3).

$$\text{Absolute error} = \left|P_{lit} - P_{meas}\right| \tag{3}$$

The physical properties (refractive index, density and viscosity) for the produced castor oil were measured compared to the literature value as presented in Table 6. The was done validate castor oil produced that is pure there is no solvent remains and absolute error was calculated.

4. Conclusion

The effect of temperature was investigated on the castor oil yield using (ethanol, furfural, d-limonene, p-cymene and *α*-pinene) as green solvents. It was revealed that green solvents produced higher castor oil yield compared to the traditional petrochemical solvent hexane. It was noted that castor oil yield increase as the temperature increases until reach optimum temperature then yield decrease, except *α*-pinene which shows a decrease in yield as temperature increases. The decreases in yield were caused by hydrolytic decomposition and rearrangement, which results in the formation of side products [33]. The green solvents were easier to recover by utilising a simple distillation. The furfural solvent was difficult to recover; therefore, it is not recommended to use in industry to replace traditional solvents such as hexane. Although, it was found that it has the highest among the selected potential green solvents. Consequently, ethanol was recommended to replace the industrial traditional solvent that affects the environment and humans.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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