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# Microwave-Assisted Extraction and Physicochemical Evaluation of Oil from *Hevea brasiliensis* Seeds

**Evelyn C. Creencia *, Joshua Andrew P. Nillama and Ivy L. Librando**

Department of Chemistry, Mindanao State University-Iligan Institute of Technology, A. Bonifacio Ave., Tibanga, Iligan City 9200, Philippines; joshuaandrewnillama@gmail.com (J.A.P.N.); ivy.librando@gmail.com (I.L.L.)
* Correspondence: ec.creencia@gmail.com; Tel.: +63-917-326-1115

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**Abstract:** The rubber tree (*Hevea brasiliensis*) is exploited mainly for latex in view of its economic importance. However, one of its auxiliary products, the rubber seed, does not find any major applications, and hence, even the natural production of seeds itself remains underutilized. In this study, microwave-assisted Soxhlet extraction is used as a green alternative to extract the oil from seeds at a reaction time of 90 min and microwave power of 300 W. The objective of the study is to evaluate the effects of the processing conditions, including drying time, temperature, solid–solvent ratio, and extraction solvent, on the yield of rubber seed oil. Moreover, the microwave-assisted aqueous extraction (MAAE) under acidic conditions is also investigated. Based on the results, n-hexane gave the best yield at an optimized 1:20 seed–hexane ratio at 72 °C compared with the conventional Soxhlet method and the acidic MAAE. Furthermore, the chemical characteristics of the oil showed a high value of free fatty acids (% FFA) (1.15–7.61%) and an iodine value (IV) that ranges from 100–150. As a semi-drying oil, rubber seed oil (RSO) can be used as an ingredient for surface coating and in the formulation of products where the presence of unsaturation is important.

**Keywords:** rubber seed oil; microwave-assisted extraction; Soxhlet extraction; microwave-assisted aqueous extraction

## 1. Introduction

The Philippine government enacted a law in 2010, Republic Act 10089, creating the Philippine Rubber Research Institute (PRRI), which is tasked with developing the rubber industry in the country. The law mandates the PRRI to "monitor and evaluate the rubber research programs and identify the immediate needs and essential concerns in the rubber industry in consonance with the local and national economic development." The PRRI is also tasked with "coordinating with other government agencies in order to formulate strategies that would jump-start the growth of the rubber industry". With the PRRI in place, the country will see more research that will investigate the development of other applications for the rubber tree.

Rubber tree (*Hevea brasiliensis*) is one of the leading commercial agricultural trees in the world, and one of the most important revenue-generating trees in the Philippines. Apart from its use in latex production for foreign exchange, the tree produces oil-bearing seeds whose oil content in dried kernel form varies from 35% to 45% [1]. Rubber seed oil (RSO) is currently being studied as a candidate component in the manufacture of various products, such as soap, paints, and alkyd resin due to its semi-dried nature. Moreover, RSO has been investigated as an effective and "green" ingredient in production of biodiesel [2]. Although the rubber seed has only been traditionally used as a planting material and is generally neglected, along with the wood from the rubber tree, studies for the utilization of the seed oil have generally increased [3]. Consequently, it is necessary to find an efficient method of extracting the oil from its natural source.

Throughout the years, numerous methods, such as expeller pressing and organic solvent extraction, have been developed in order to extract oil from seed materials [4]. However, regulatory problems associated with the use and disposal of organic solvents have undesirable effects on oil and costs [5]. Driven by these purposes, advances in sample preparation have resulted in a number of techniques that both improve the extraction yield, as well as minimize or even eradicate the drawbacks posed by conventional techniques.

Microwave-assisted extraction (MAE) is a novel technology, which has been studied extensively in recent years, mainly to reduce extraction time and solvent consumption [6,7]. This new technology involves lower energy consumption, higher extraction yields, and less production of waste, which results in a more economically favorable extraction procedure [8]. It has been well-documented that microwave treatment of oilseeds during solvent extraction leads to the protein denaturation of cells, resulting in an improved extraction [9]. Furthermore, microwave-assisted extraction can also reduce the use of organic solvents, because it can generally make use of aqueous solvents as the extraction media. This technique is specifically called microwave-assisted aqueous extraction (MAAE).

Thus, this study will put forward the use of microwave-assisted extraction as a green alternative method for the extraction of rubber seed oil, as well as the evaluation of its physicochemical properties in terms of appearance, free fatty acid content, iodine value, and peroxide value. In addition, this study will also be able to promote the valorization of rubber seeds, as well as its utilization as a renewable source to produce seed oil, which can be used for a variety of applications, particularly to produce biofuel.

## 2. Materials and Methods

### *2.1. Materials and Reagents*

Fresh rubber (*Hevea brasiliensis*) seeds were collected from rubber plantations in Zamboanga Sibugay, Philippines (7°47′24.19″ N, 122°35′37.78″ E). The seeds were subjected to shell/husk removal in order to separate the kernel. The seeds were cut into smaller pieces and oven-dried at 105 °C for 2, 3, 4, and 18 h, respectively. Then, the dried kernels were ground to fine powder (40-mesh) for oil extraction. All chemicals and reagents were purchased commercially, of analytical grade, and were used as received.

### *2.2. Microwave-Assisted Soxhlet Extraction*

Five g of ground rubber seeds were placed into the extractor through a thimble, and the oil was extracted with 100 mL of four different solvents (n-hexane, n-hexane–acetone (3:1 $v/v$), n-hexane–ethanol (3:1 $v/v$), and absolute ethanol) for 90 min at a power of 300 W using the Milestone RotoSYNTH microwave reactor (Milestone Srl, Bergamo, Italy). Low-polar and nonpolar extractants were heated to their boiling points while stirring with a Weflon magnetic bar (Milestone Srl, Bergamo, Italy) to aid absorption of microwave radiation. After extraction, the extract was filtered to remove particles that were entrained during the process. The solvent was separated from the crude rubber seed oil using a rotary evaporator.

The oil yield obtained was expressed in terms of mass percentage of the samples and calculated as follows:

$$\text{Oil yield (wt \%)} = \frac{\text{mass of oil extract (g)}}{\text{mass of ground rubber seed (g)}} \times 100 \quad (1)$$

### *2.3. Acidic pH-Based Microwave-Assisted Aqueous Extraction*

About five g of ground rubber seeds were placed into the extracting flask through a thimble and added with a specified volume of the extracting solvent ($1 \times 10^{-3}$ M HCl, $1 \times 10^{-4}$ M HCl, $1 \times 10^{-6}$ M HCl, and distilled water) to produce a solid–solvent ratio of 1:12 $m/v$. The initial temperature and time were then set based on the results from the performed optimization.

The microwave power was also set to 500 W, and the stirring was kept at 70%. After the extraction, the sample was removed from the flask and heated in a boiling water bath for 15 min for demulsification. After cooling to room temperature, the mixture was then washed twice with 15-mL portions of n-hexane, producing an aqueous layer and a foam layer. The obtained foam layer was afterwards subjected to the rotary evaporator and centrifuged at 3000 rpm for 30 min to completely break the layer, producing an aqueous phase and an upper organic phase. The organic phase was then collected and subjected to the rotary evaporator to recover the extracted seed oil, which was then dried to constant weight. The oil yield was then calculated using Equation (1).

### *2.4. Conventional Soxhlet Extraction*

The conventional Soxhlet extraction was performed using the official method for oil extraction from the Association of Official Analytical Chemists (AOAC, 1997) [10] with few modifications. About 10 g of ground rubber seeds were placed into the extractor through a thimble, and the oil was extracted with approximately 120 mL of n-hexane. The extraction process was allowed to progress for 6 h, and the solvent–oil mixture was filtered and subjected to solvent removal by rotary evaporator to recover the crude oil extract.

### *2.5. Evaluation of Physicochemical Properties*

The characterization of the obtained seed oils was performed using the American Oil Chemists' Society (AOCS) standard methods.

- Free fatty acid (FFA)—The FFA content was evaluated using AOCS Official Method Ca 5a-40.
- Iodine Value (IV)—The IV was carried out following the AOCS Official Method Cd 1d-92.
- Peroxide Value (PV)—The PV was measured following the AOCS Official Method Cd 8b-90.

## 3. Results and Discussion

### *3.1. Microwave-Assisted Soxhlet Extraction*

Observations have been made regarding the physical characteristics of rubber (*Hevea brasiliensis*) seed oil extracted from the four different solvents. The oil from n-hexane was clear and golden yellow in color, with the original odor of the rubber seed. This is because n-hexane has low polarity, which is miscible in oil and extracts only the glycerides in the seeds. On the other hand, white particles were observed to be extracted together with the oil upon the addition of a polar solvent to n-hexane. Hron et al. [11] and Ferriera-Dias et al. [12] reported that polar solvents, such as ethanol, have the capability to co-extract other polar compounds in oilseeds, which are insoluble in non-polar solvents. The oil extracted using absolute ethanol was viscous, yellowish to brownish in color with an emulsion-like appearance.

#### 3.1.1. Effect of Solid–Solvent Ratio on Oil Yield

In this study, three mass–volume ratios of seeds to solvent (n-hexane) were tested using the 18-h oven-dried, ground rubber seeds. As shown in Figure 1, the highest oil yield was obtained at the 1:20 solid–solvent ratio. Therefore, it can be said that increasing the ratio also increases the yield. This is because the concentration gradient between the solid and the liquid phase becomes greater, which favors good mass transfer and increased extraction efficiency [13]. Subsequent experiments on microwave-assisted Soxhlet extraction were carried out using the 1:20 ratio.

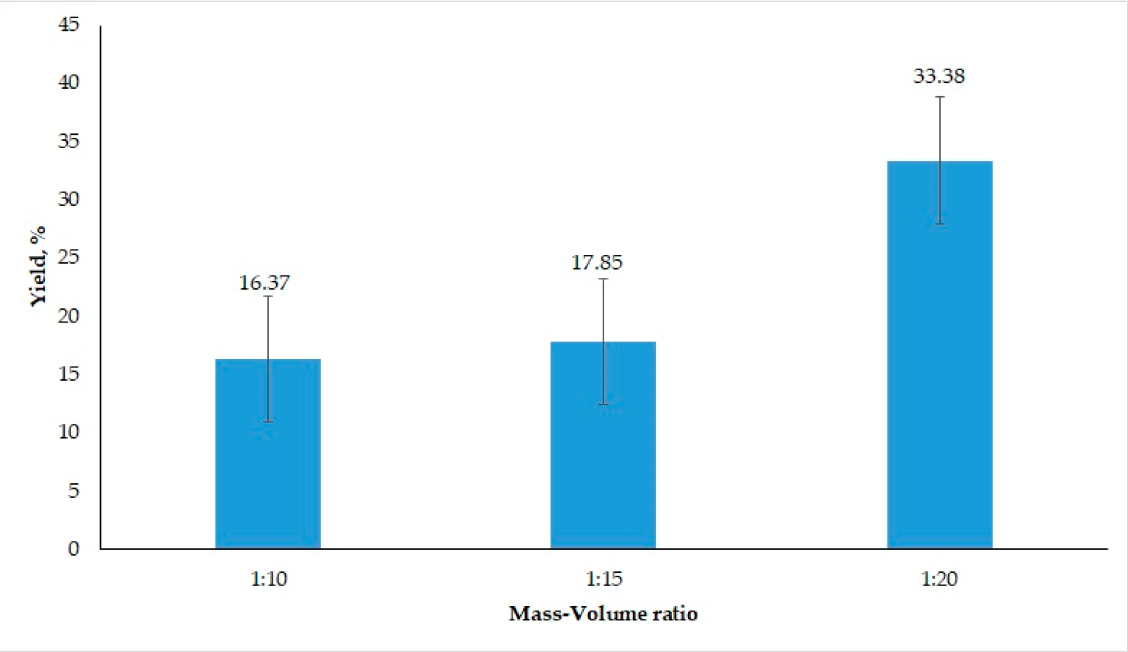


**Figure 1.** Effect of mass–volume (n-hexane) ratio on the extraction efficiency with a reaction time of 90 min at 72 °C and microwave power of 300 W.

### 3.1.2. Influence of Oven-Drying Time

The effect of oven-drying the sample at 2, 4, and 18 h on the oil yield is graphically illustrated in Figure 2. It is observed that, irrespective of the solvent used, high oil yields were obtained at the shortest oven-drying time, which was the 2-h period. This result can be attributed to the moisture content of the ground rubber seeds prior to extraction. Previous studies have shown that moisture in seeds plays a significant role during oil extraction. Accordingly, protein coagulation is promoted as heat is transferred through the moisture in the oilseeds, and this has been shown to facilitate the release of oil. Moreover, as the drying time of the samples is increased, so is moisture loss. Relatively long drying times are therefore associated with harder seed particles as a result of the very low moisture content after heating. This consequently causes the oil to be more attached to the seeds and hinders its release, thereby lowering the oil extraction yield [14]. This may account for the reduction in oil yield for the 18-h, oven-drying period.

### 3.1.3. Influence of Different Extraction Solvents

In the method used, four different extraction solvents of varying polarities were used. The low-polar solvents were n-hexane, n-hexane–acetone (3:1 $v/v$), and n-hexane–ethanol (3:1 $v/v$), while the polar solvent used was absolute ethanol. Because the extracted RSO is not intended for human consumption, but rather for other industrial applications, particularly biofuel production, the use of the aforementioned solvents was considered safe for this utilization. Furthermore, the purpose of using solvents with different polarities was to investigate the influence of solvent polarity on the yield and the physicochemical characteristics of the oil. As reported in Figure 2, the yield of oil extracted by n-hexane was the highest despite being dependent only on the Weflon magnetic bar to absorb microwave radiation due to its nonpolar nature. This suggests that nonpolar rubber seed oil is easier to be extracted by a nonpolar solvent such as n-hexane. Even though absolute EtOH absorbs microwave radiation efficiently, the polar nature of this solvent conceivably destroys intracellular compartmentalization in the seeds, allowing solubilization of more unsaponifiable matter and leading to lower oil yields [15].

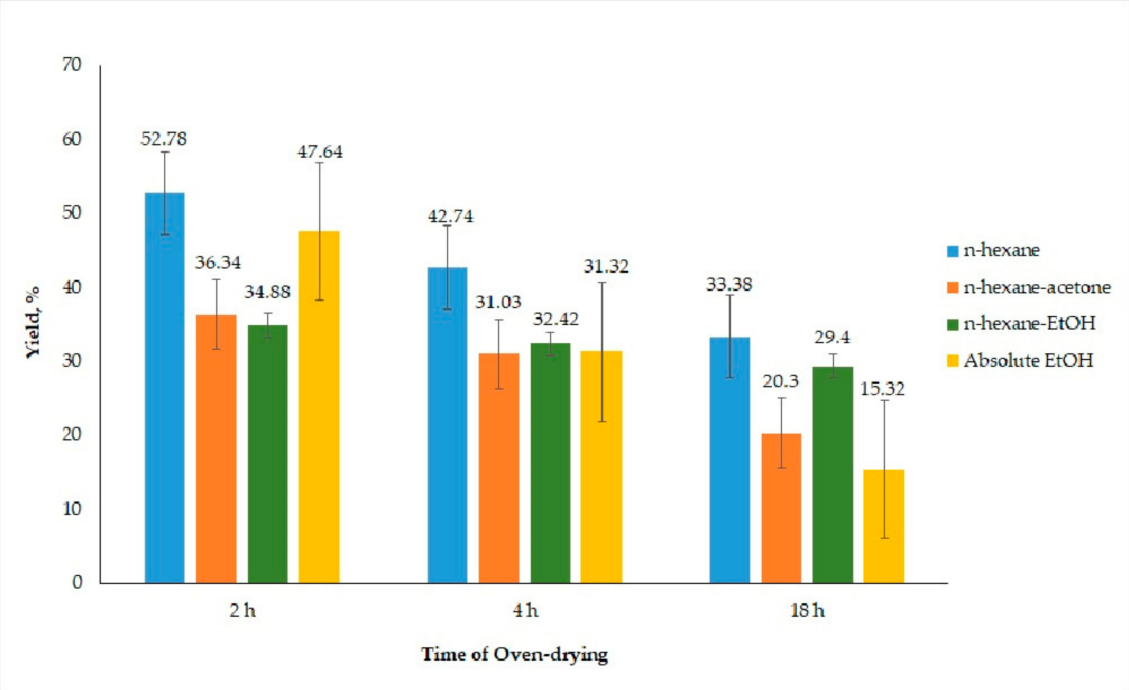


**Figure 2.** Variation of oil yields at 2, 4, and 18 h of oven-drying time with a reaction time of 90 min and microwave power of 300 W.

### 3.1.4. Influence of Temperature

The effect of the boiling temperature of solvents on the yield is presented in Figure 3. The optimum oil yield was obtained at 72 °C, which corresponds to the boiling temperature of n-hexane. It is observed that oil yield increased initially with increasing temperature. However, the continuous increase in temperature resulted in the decrease in attained oil yields. These results can be accounted for by the idea that at elevated temperatures, the disruption of the cell walls in oil-bearing cells can be promoted. This allows the oil content in the cells to easily escape from the cells into the extracting solvent, which increases the extraction yield. However, upon doing the extraction at even more elevated temperatures, the degradation of the oil is very much likely to occur. Moreover, the samples can lose a very significant amount of their moisture content. This, as mentioned previously, leads to seed hardening and subsequently, to lower extraction yields [16–18]. This may account for the decrease in oil yield from 70 to 80 °C.

Figure 4 shows the oil yields of the MW-assisted extraction method using the 2 h oven-dried samples with four different extraction solvents and irradiated at 300 W for 90 min, while the oil yield for the conventional Soxhlet extraction was obtained from a 2 h oven-dried sample with n-hexane, extracted for 6 h. The data presented shows that the yield obtained from the MW-assisted extractions using n-hexane and absolute EtOH as solvents gave higher yields than the conventional Soxhlet extraction. This signifies that the MW-assisted extraction is more efficient in terms of oil yield and time consumption as it can achieve a maximum extraction within 90 min, which cannot be achieved through the conventional Soxhlet method even after 6 h of extraction.

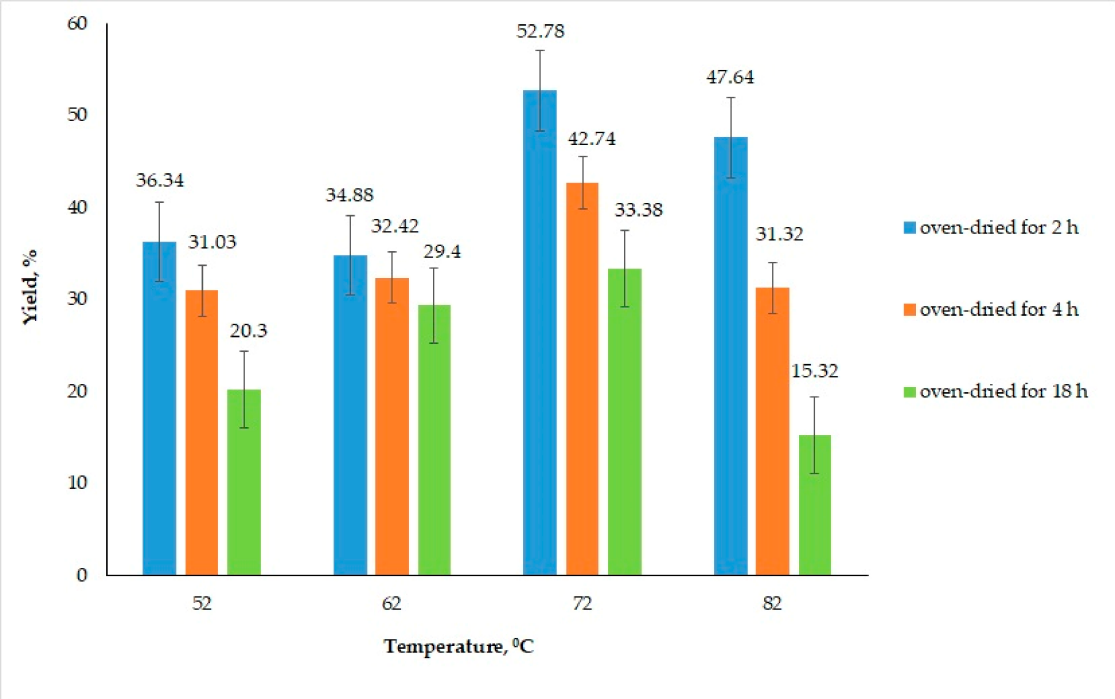


**Figure 3.** Effect of temperature on the extraction efficiency with reaction time of 90 min and microwave power of 300 W.

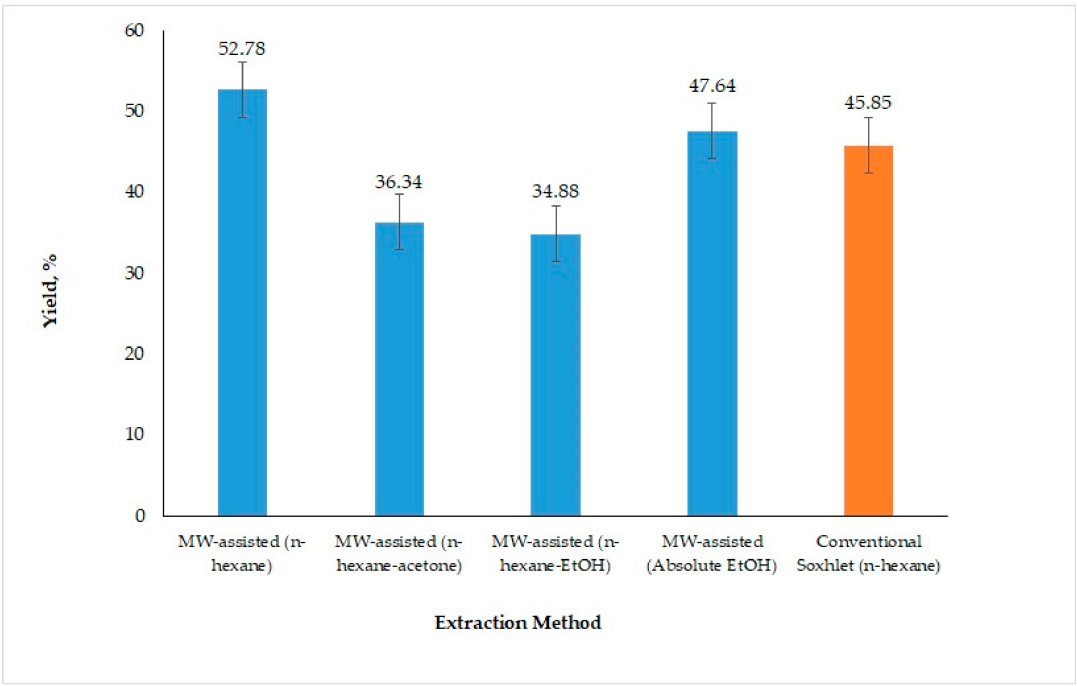


**Figure 4.** Comparison of the oil yields for the MW-assisted (oven-dried for 2 h) and the conventional Soxhlet extraction methods.

### *3.2. Acidic pH-Based Microwave-Assisted Aqueous Extraction*

Aqueous extraction of rubber seed oil under the influence of microwave radiation was carried out after performing the necessary optimization procedures for the selected extraction parameters, namely extraction temperature and time. The best conditions were then identified from these optimizations

and used for the extraction procedure. Moreover, due to the aqueous nature of the extracting solvent used for the acidic MAAE, the solid–solvent ratio was reduced to 1:12 (g/mL).

#### 3.2.1. Determination of Best Conditions for Oil Extraction

From the results presented in Figure 5, it can be seen that the highest extraction yield, with a value of 10.09%, was attained using the temperature setting of 80 °C. A general trend of increasing oil extractability with increasing temperature can be observed as the temperature is elevated from 60 °C to 80 °C. However, it can also be inferred from the data that only a very small increase in extraction yield can be observed as the temperature increases within the stated range. This is very evident in the 60 °C and 70 °C settings where the extraction yield remained at approximately 8%. This is an indication that within this range, temperature changes do not have any significant effect on the oil yield. In addition, since 90 °C is substantially close to the boiling point of water, which is the main component of the extracting solvent, gradual evaporation of the solvent may have taken place to a considerable extent as the extraction progressed. This can also be considered as a contributing factor to the decrease in the extractability of the seed oil, because this alters the initial solid–liquid ratio of the system.

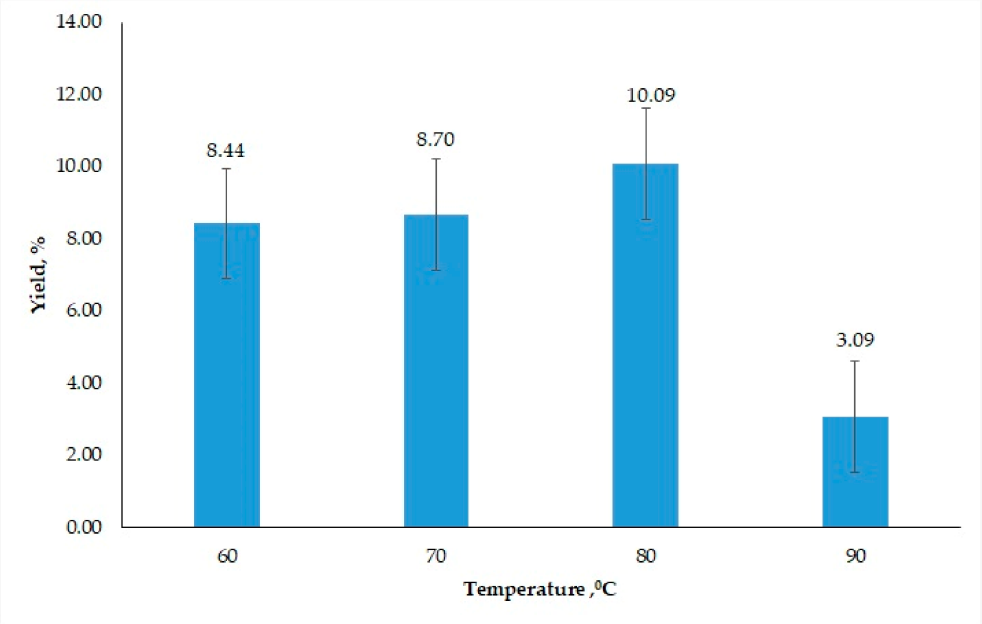


**Figure 5.** Variation of extraction yield with temperature.

Nonetheless, because the temperature setting of 80 °C gave the best results, this setting was used in the succeeding optimization for extraction time. As shown in Figure 6, it is very evident that carrying out the extraction for 90 min gave the highest oil yield, with a value of 10.09%. This suggests that exposure of the sample to the microwave irradiation at this time interval caused a relatively sufficient disruption of the cell walls, thereby allowing better release of the oil from the sample cells. At a shorter extraction time (60 min), the length of the exposure of the sample to the microwave radiation was not enough to cause a complete disruption of the cell walls and hence, the ineffective release of oil. Furthermore, lowest extraction yield was obtained at the 150-min extraction time. This may be due to emulsification promoted by the longer contact of the released oil with water-soluble proteins in the seed, which exhibit surfactant activity upon denaturation.

The extraction temperature and time were set at 80 °C and 90 min, respectively, since these settings gave the highest yield in the preceding optimization procedures. The acidity of the extracting solvent was regulated from pH 3 to pH 7 using diluted hydrochloric acid for solutions of pH 3–6 and commercial distilled water for pH 7. The effect of pH on oil extractability is shown in Figure 7.

The general trend that can be observed from the data is that the % yield increases with the increasing pH of the extracting solvent until pH 5. At a lower pH, lower extraction yields were observed, which may be due to proteins occurring at the isoelectric point, which aggregate with oil bodies, hindering the release of oil [4]. Beyond pH 5, a decrease in oil extractability was also observed. This is primarily due to the lesser amount of acidic species in the solvent for these pH, which means that only few acidic species can participate in the disruption of the oleosins covering the oil bodies in the seeds, and therefore, ineffective release of oil occurs.

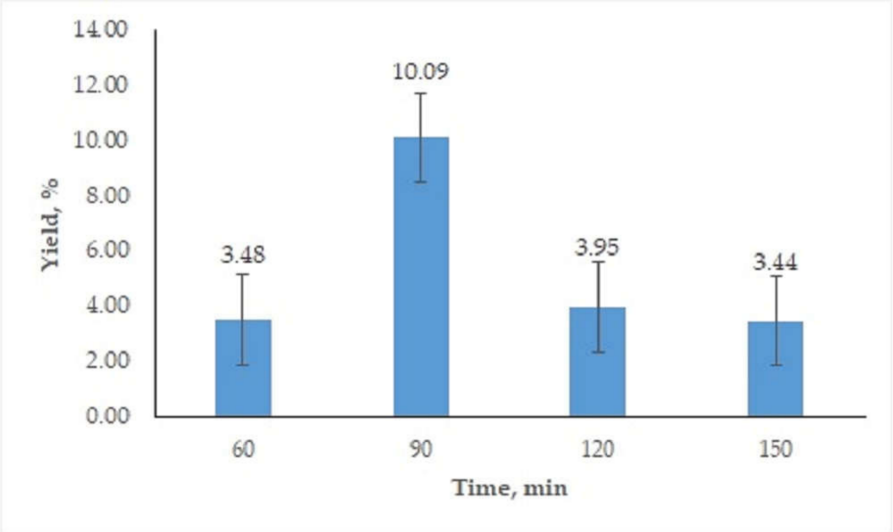


**Figure 6.** Variation of extraction yield with time.

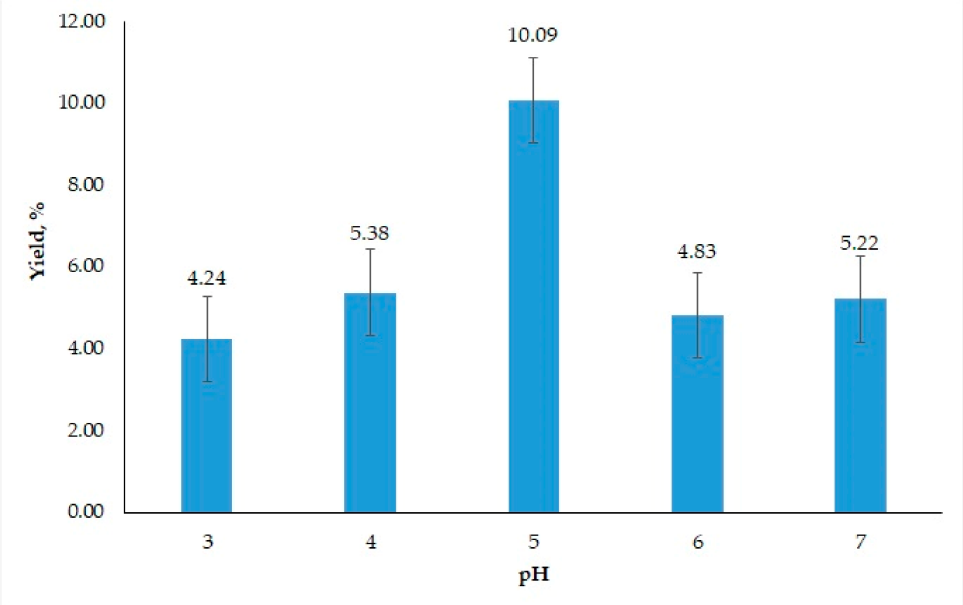


**Figure 7.** Variation of the extraction yield with pH of the extracting solvent.

### 3.2.2. Comparison of Acidic pH-Based MAAE and Conventional Soxhlet Method

It is evident from the data presented in Figure 8 that the extraction yield for the acidic pH-based MAAE is only about 25% of the extraction yield for the conventional method. This signifies that the

effect of an acidic environment was not enough in preventing the aggregation of proteins with the oil during extraction.

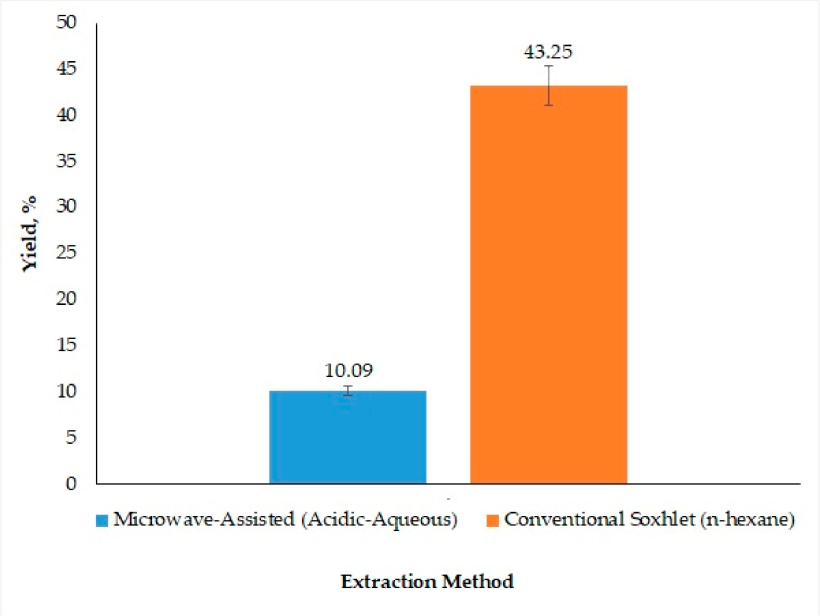


**Figure 8.** Comparison of extraction yields for the acidic pH-based microwave-assisted aqueous extraction (MAAE) and conventional Soxhlet method.

Moreover, this significantly large difference in extraction yields can be attributed to the fact that the determination of the best extraction conditions was only limited to the optimization of two extraction parameters. In the method used, only the effect of temperature and time were examined. As a result, the possible contributions of the other parameters in the extractability of the sample were no longer accommodated, which may have served as a contributing factor to the observed smaller yield for the MAAE. Furthermore, from these results, it can be inferred that in terms of extraction yield, the conventional method was a more effective technique.

### *3.3. Evaluation of Physicochemical Properties*

The physicochemical properties of seed oils obtained from the MW-assisted Soxhlet extraction, the acidic pH-based MAAE, and the conventional Soxhlet methods were evaluated in order to compare their quality. The results for these determinations are summarized in Tables 1 and 2.

**Table 1.** Physicochemical properties of the extracted *Hevea brasiliensis* seed oil.

| Parameter | Conventional Soxhlet Method [b] | MW-Assisted Soxhlet Extraction | | | | | |
|---|---|---|---|---|---|---|---|
| | | n-Hexane | | | n-Hexane–Acetone (3:1 *v*/*v*) [a] | n-Hexane–Ethanol (3:1 *v*/*v*) [a] | Absolute Ethanol [a] |
| | | 2 h | 4 h | 18 h | | | |
| FFA (% Oleic) | 7.61 | 5.16 | 3.91 | 1.19 | 5.43 | 5.20 | 3.67 |
| Iodine Value | 132.8 | 107.7 | 119.9 | 121.9 | 150.2 | 148.3 | 147.7 |
| Peroxide Value (mEq/kg) | 15.13 | b | b | b | b | b | b |

[a] Oven-dried for 2 h; [b] Cannot be determined.

**Table 2.** Physicochemical properties of the extracted *Hevea brasiliensis* seed oil (oven-dried for 3 h).

| Parameter | Conventional Soxhlet Method | Acidic pH-Based MAAE | | |
|---|---|---|---|---|
| | | pH 3 | pH 5 | pH 7 |
| FFA (% Oleic) | 2.54 | 1.66 | 1.15 | 1.64 |
| Iodine Value | 131.9 | 131.2 | 126.4 | 129.7 |
| Peroxide Value (mEq/kg) | [b] | 19.50 | 5.18 | 5.62 |

[b] Cannot be determined.

From the results, it is observed that the seed oil extracted using the conventional Soxhlet method exhibited a higher FFA value compared with the oil extracted using the MW-assisted methods. The higher FFA content obtained from the conventional Soxhlet method implies that this oil has a higher tendency of becoming rancid than the oil extracted from the MAE. Factors, such as longer time of extraction (6 h) and no control of temperature in a conventional extraction, may have reinforced each other in promoting the hydrolysis of the triacylglycerols in the oil to occur, thereby causing greater FFA results. On the other hand, as shown in Table 1, the values of free fatty acid decreased with an increase in the oven-drying time of the seed from 2 h to 18 h. This means that the longer the oven-drying time of the seeds, agents of rancidity, such as moisture, are much reduced than at a shorter oven-drying time.

The iodine value is often used to assess the drying property of oils. The IV of the samples ranges from 100–150 as shown in Tables 1 and 2, indicating that the oil samples contain high amounts of unsaturated fatty acids. Moreover, these results verify the semi-drying property of RSO, which signifies that it may be a potential ingredient in the formulation of surface coatings. According to Warra et al. [19], oils with IVs that fall within the 100–150 range display the ability to absorb oxygen when exposed to air, which thickens them and allows them to remain sticky without forming a hard, dry film. In addition, unsaturation in vegetable oil is a desirable property in vulcanized oil synthesis. Thus, the high IV recorded for RSO is a strong indication that it would be suitable for vulcanized oil synthesis.

The observed high PV for RSO (19.50 and 15.13 mEq/kg) may be attributed to the heating during extraction, as heat favors the oxidation of fatty acids and most especially polyunsaturated fatty acids. The lower values of PV (5.18 and 5.62 mEq/kg) imply that these oils have lower degree of rancidity. From the results given, the PV of some of the samples could not be determined because they were unable to produce a deep blue color upon the addition of a starch indicator; thus, they could not be titrated to their endpoints. This suggests that only small amounts of peroxides were present in these samples.

## 4. Conclusions

The extraction of oil from *Hevea brasiliensis* seeds was investigated using microwave-assisted extraction methods. In general, n-hexane gave the best yield at an optimized 1:20 seed–hexane ratio at 72 °C. This method is more efficient in terms of yield and time consumption as it can achieve a maximum extraction within 90 min, which is much faster than the conventional Soxhlet method that requires 6 h of extraction. The results also showed that extraction using MAAE under acidic conditions is best carried out at 80 °C for 90 min at pH 5. However, the results revealed that the extraction yield for this method was only 25% of the conventional Soxhlet method. This signifies that the conditions used for this method are not sufficient to prevent the aggregation of the oil with seed proteins. From the obtained results for chemical analysis, the high iodine and FFA values for rubber seed oil serve as an indication of its potential as an ingredient in soap and surface coatings, as well as in biodiesel and vulcanized vegetable oil (VVO).

Overall, the method presented in this study was found to be more efficient due to the higher oil extraction yield of 52%, which only required 90 min, compared with other techniques for RSO extraction, which required longer extraction time but produced lower yields [3]. Aside from

demonstrating the opportunity to use MAE to replace the time-consuming conventional extraction process, the research findings also generated new and vital information for the exploitation of the capability of the rubber seed as a promising raw material for oleochemical processes. This, in turn, can be a helpful reference for the rubber seed agriculture industry in expanding profit. Moreover, the method used in this study may be used as a reference for future research studies that focus on RSO extraction particularly for biodiesel applications. Other possible future developments for the study could also include the refinement of the extracted RSO using methods such as esterification, as well as the assessment of the chemical properties of the biodiesel produced from the refined RSO.

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**Author Contributions:** The authors contributed equally to this work.

**Conflicts of Interest:** The authors declare no conflict of interest.

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