COMPARATIVE STUDY BETWEEN MICROWAVE ASSISTED EXTRACTION AND SOXHLET EXTRACTION TECHNIQUES FOR BIO-OIL EXTRACTION FROM JATROPHA CURCAS

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Abstract

Microwave Assisted Extraction (MAE) was used to obtain crude oil from Jatropha curcas seeds. The sample were extracted at different volume of water that act as solvent (50 mL, 100 mL, 150 mL and 200 mL). The oil yield obtained was compared with the Soxhlet Extraction (SE) oil yield. The yield from MAE at 50 mL, 100 mL, 150 mL, and 200 mL was 19%, 31%, 33%, and 40% respectively. The aromatic compound was present in the MAE extracted oil but was not found in SE extracted oil. There is a major disruption in alkanes group as sample was analysed before and after extraction. MAE is more favourable compared to SE as it do not consumed hazardous organic solvent, low cost and has extremely shorter extraction time compared to SE.

Keywords: Jatropha oil; FTIR; Microwave assisted extraction.

1. Introduction

Jatropha curcas was once considered in valuable and unuseful as its produce inedible oil and contained toxic compound such as phorbol esters. As the need to cater the world demand for biodiesel, along with other inedible oil plants, it has once again become popular sources. Jatropha curcas (Linnaeus) belongs to Euphorbiaceae family.

Jatropha is commonly known as Jamalghota. Jatropha plants originated from North American and now are abundant in country such as Malaysia, Thailand, Mexico, China, Ghana, Mali Nigeria, Indonesia, South Africa, Haiti, and Congo [1-6].

This plant has been used as fences for houses or farms and easily grows on abandoned land [2-3, 7]. Jatropha curcas is a drought resistant tropical tree and
the oils contain in its seed has many benefits with wide applications such as for medicinal purposes, veterinary, as insecticide, soap production and also biodiesel [8].

In Brazil, it has widely been used as an insecticide for disinfecting houses against household bugs. Their bark is believed to play crucial role in preventing cancer cell from spreading. The scraping from the bark can be utilised in the production of blue coloured dye [6]. Jatropha curcas is easily grown in poor soil conditions with low sources of water and nutrient thus preventing soil erosion.

Jatropha curcas seed contain inedible oil which is about 30-35% [4]. Other work [9] has reported that Jatropha curcas seeds contain around 20-40% oil. Oil extracted from Jatropha curcas seed have concentration ranging from 47-50% using Soxhlet Extraction [5]. It has been reported seed oil content based on a dry basis was 46% [10]. The oil content in Jatropha curcas seed is 25-30% and compose of 21% saturated fatty acids and 79% unsaturated fatty acid [5].

Oil in Jatropha curcas seed usually extract by conventional method such as Soxhlet Extraction, expellers, mechanical press as well as chemically and enzymatically. As consumption of organic solvent in widely used solvent extraction is hazardous, research interest in green technology which is solvent free extraction technique is becoming very important. Pressurize Hot Water Extraction, Supercritical Carbon Dioxide, Ultrasonic Extraction Technique as well as Microwave Assisted Extraction are considered as green technology and advantages over conventional method in term of shorter extraction time, no consumptions of hazardous organic solvent and not hazardous to human.

This research intends to explore the application of Microwave Assisted Extraction which is solvent free extraction technique in extraction of oil from Jatropha curcas seed. Another extraction method used was soxhlet extraction as to compare the oil obtained using solvent free extraction with oil extract using conventional method.

2. Material and Methods

2.1. Materials

*Jatropha curcas* seeds were collected from University Malaysia Perlis (UNIMAP) Agrotechnology Centre. Equipment employed in this research include drying oven, kitchen blender, Moisture Analyzer Sartorius, mortar and pestle, round bottom flask, analytical balance, vacuum pump, Buchner filtration assemble, sieve, rotary evaporator, Soxhlet Extractor, Sharp microwave attach with condenser and round bottom flask. Chemical used is hexane for extraction. Chemical compound identification was determined by using Fourier Transform Infrared (FTIR).

2.2. Methods

2.2.1. Sample preparation

*Jatropha curcas* seeds were collected manually from UNIMAP Agrotechnology Centre. The seeds were dehulled to remove shell afterward the seeds dried in oven at 40°C for 24 hours. The sample then ground using kitchen blender until desired
particle size is obtained. The ground seeds were sieved to particle size of 180 µm - 500 µm. The sample was stored in container before further experiment.

2.2.2. Soxhlet extraction (SE)
100 g of ground sample was placed into thimble and extracted using 350 mL of hexane in a soxhlet extractor for 8 hours. The yield was collected by separating the solvent using rotary evaporator (Buchi R210) and remaining solvent was evaporate off in drying oven at 60 °C for 1 h.

2.2.3. Microwave assisted extraction (MAE)
10 g of Jatropha curcas seeds were placed in 250 mL round bottom flask. The solvent used is distilled water (dw). The oil was extracted using four different amount of solvent which is 50 mL, 100 mL, 150 mL, and 200 mL for 1 hour at 700 watt microwave power. The solvent was mixed with sample in a round bottom flask attached with condenser. The condenser was connected to the ice box containing ice pack, vacuum pump, ice cube and water as shown in Fig. 1. Water, oil and seeds cake was separated by filtration using Buchner Filtration assemble. Oil and water separated using rotary evaporator (Buchi R210) using oil bath at 110 °C.

Fig. 1. Microwave assisted extraction equipment.

2.2.4. FTIR analysis
Four samples were subjected for FTIR analysis which are ground Jatropha curcas seeds, seeds cake, oil extracted using Soxhlet Extractor and oil extracted using MAE. The analysis was performed using PERKIN ELMER SPECTRUM ONE model instrument at wave number ranging from 350-4000 cm⁻¹.

3. Results and Discussion
3.1. Extraction of Jatropha curcas using MAE and SE
Oil extracted from both methods was compared in term of their respective yield (w/w %) in Table 1. Oil yield from Soxhlet extraction for 8 hours using Hexane is
48%. This result was slightly higher than 46% reported by Joshi et al. [10]. Oil extracted from MAE for 1 hour using different amount of solvent are 19%, 31%, 33% and 40% for 10 g/50 mL, 10 g/100 mL, 10 g/150 mL and 10g/200 mL respectively. The volume of solvent used was up to 200 mL in order for the reaction to occur smoothly inside 250 mL round bottom flask.

Extraction of *Jatropha curcas* oil using MAE has advantages over conventional extraction method as it does not consume hazardous organic solvent as well as environmental friendly. Extraction occurs at shorter reaction time which is 1 hour compared to extraction using Soxhlet Extractor which was 8 hours extraction time which is not cost effective and environmental friendly.

200 mL of water was used to produce 40% oil yield. Compared 48% oil yield was obtained with 350 mL hexane and 8 hours extraction time. Therefore MAE is an effective and green technology of producing bio-oil from *Jatropha curcas*. The longer reaction time taken in soxhlet extraction in order to yield 48% oil was due to conventional heating process that is much slower compared to microwave heating process. Microwave energy work selectively with water surrounded the sample as well as reacted with polar molecule in form of moisture inside cell that cause cell wall expansion. The expansions permit force and induce the breaking of cell wall thus releasing oil to the outer environment.

The 40% oil yield from microwave assisted extraction can be increase to the same or even higher amount of oil yield obtain by Soxhlet extraction with few modification to the experiment such as additional amount of solvent, use higher power of microwave energy (watt), increase extraction time, additional of boiling chips to enhance mixture reaction or by using stirrer to ensure the uniformity of mass transfer between water and sample.

The higher amount of yield obtain using soxhlet was due to the used of hexane that is soluble with oil which aid the release and mass transfer properties of oil compared with water that is less soluble with oil. Rapid heating mechanism occurred in microwave has assist the mass transfer process as well as penetration of water into sample. As microwave work selectively with water, microwave energy react with water molecule inside sample which facilitate and improve the cell wall rupturing.

From Table 1, volumes of solvent influence the yield percentage in MAE extraction process. The percentages of yield correspond to the amount of water used 50 mL < 100 mL < 150 mL < 200 mL. Higher amount of water help to absorb the energy supply by MAE thus increasing the reaction occurred between solvent and sample thus resulting in higher temperature which enables the penetration of water into sample. This will trigger the release of oil from sample’s matrix at shorter reaction time.

3.2. FTIR analysis

FTIR analysis was conducted on four different samples which are *Jatropha curcas* powder before extraction, *Jatropha curcas* seed cake after extraction, *Jatropha curcas* oil extracted using SE and *Jatropha curcas* oil extracted using MAE. The entire bio-functional groups were as shown in Table 2 and Figs. 2-5.
There were significant changes of *Jatropha curcas* seed before and after extraction using MAE, Figs. 4(a) and (b). The alkanes group disappeared after MAE while there was new bond form after MAE which was the alcohol and the phenol group. This is caused by the heating process which took place during extraction involving a direct heating that was selective towards sample. The reaction could have caused the disruption of alkanes bond in the sample which assists the penetration of solvent into sample as well as discretion of oil.

Oil from SE as shown in Fig. 3 only has two bio-functional groups which were alkanes and alcohol group while there were four bio-functional groups present in oil extracted using MAE listed as alkanes with C-H stretching and bending, alcohol and aromatic compound in Fig. 2. The advantage of extracting oil using MAE is that the present aromatic compound after extraction was retained while in oil extracted using SE the bonds were disrupted.

In this study it was shown in Fig. 2 and Table 2 that MAE was able to enhance the number of chemical compound and also yield depending on the amount of water used during extraction. The used of water in extraction technique has produced an effective products without being chemically modified as SE.

The chemical compounds present can be isolated and processed according to its specific use as the chemical compounds can serve as starting compound in the synthesis of other value added products. By analysing the chemical and functional group present, the use of *Jatropha curcas* waste after extraction can be reutilized as it has high potential to serve as biofertilizer, fish feed, or for medicinal purposes.

![Fig. 2. Chemical compound present in Jatropha Curcas oil extracted using MAE.](image)

**Table 1. Jatropha curcas Oil Yield (%w/w).**

<table>
<thead>
<tr>
<th>Solvent volume (mL)</th>
<th>Yield percentage (%w/w)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Microwave 50</td>
<td>19.25</td>
</tr>
<tr>
<td>Assisted 100</td>
<td>31.00</td>
</tr>
<tr>
<td>Extraction 150</td>
<td>33.10</td>
</tr>
<tr>
<td></td>
<td>200</td>
</tr>
<tr>
<td>Soxhlet Hexane</td>
<td>48.16</td>
</tr>
<tr>
<td>Extraction 350 mL</td>
<td></td>
</tr>
</tbody>
</table>

*Results are based on triplicate average value of sample with standard variation of ± 1.5.*
Table 2. Comparisons of bio-functional group of *Jatropha Curcas* powder and oil.

<table>
<thead>
<tr>
<th>Wave number range</th>
<th>Group</th>
<th>Compound</th>
<th>Sample before extraction</th>
<th>Sample after extraction</th>
<th>Oil from SE</th>
<th>Oil from MAE</th>
</tr>
</thead>
<tbody>
<tr>
<td>3600-3200</td>
<td>O-H</td>
<td>Alcohol Phenol</td>
<td>-</td>
<td>X</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>3000-2850</td>
<td>C-H</td>
<td>Alkanes</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>3000-1000</td>
<td>C-O</td>
<td>Alcohol Carboxylic acid</td>
<td>X</td>
<td>X</td>
<td>X</td>
<td>X</td>
</tr>
<tr>
<td>1680-1600</td>
<td>C=C</td>
<td>Alkenes</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1450-1375</td>
<td>C-H</td>
<td>Alkanes</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>960-600</td>
<td>C-H</td>
<td>Aromatics</td>
<td></td>
<td>X</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Fig. 3. Chemical compound present in *Jatropha Curcas* oil extracted using SE.

Fig. 4(a). Chemical compound present in *Jatropha Curcas* powder before extraction using MAE.
Comparative Studies Between Microwave Assisted Extraction and Soxhlet

4. Conclusion

Microwave Assisted Extraction has proved to have shorter reaction time compared to Soxhlet Extraction and having similar yield which is 40% and 48% respectively. Further work will be done in order to achieve higher oil yield obtain from microwave assisted extraction process with additional modification to the process or parameters involve in reaction process. Microwave assisted extraction is a green technology where only water is used compared to Soxhlet extraction that utilize hazardous organic solvent that bring acute effect toward our health and environment. The bio-oil produced from Microwave assisted extraction technique is of high quality as denoted by the compound present. It is not only a green technique but it is also fast and cost effective techniques as it used water substitute for other hazardous organic solvent. Water is readily available, cheap and abundant and faster heating process prompt by microwave energy has save hours of time and immense amount of energy. This research is important as it demonstrate the opportunity of using MAE to replace time consuming conventional extraction process. The next step to be taken is developing MAE in pilot scale to study its efficiency with higher amount of sample than this lab scale study.
Acknowledgements
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References