Determination of Oil Content in Biodiesel Feedstock by Accelerated Solvent Extraction

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INTRODUCTION

The determination of oil content in biodiesel feedstock can be performed by several methods, including mechanical press, solvent extraction, and Nuclear Magnetic Resonance (NMR). For feedstock quality control in terms of oil content, it is important that the applied method is universally accepted so that the results can be compared with those from different sources. Although the European standard has specified two methods for the determination of oil content in oilseeds, conventional Soxhlet extraction and NMR imaging, these methods have disadvantages, including excessive time consumption, labor-intensive input, need for more highly skilled labor, a requirement for significant number of samples, high cost, and being harmful to the environment.

The Accelerated Solvent Extraction (ASE®) method developed by Dionex has great potential in overcoming these constraints. Research by the Institute of Environmental Science and Engineering in Singapore evaluated this potential. The results demonstrate that the ASE method can be used efficiently to extract oil from Jatropha seeds with comparable results, in terms of accuracy and repeatability, to the results obtained from the European standard. The oil extraction using the ASE method requires less than 1 h compared to the 10 h consumed by the Soxhlet extraction method (Table 1). The ASE method can also be used effectively with smaller sample sizes.

EQUIPMENT

- ASE Accelerated Solvent Extractor equipped with 11 or 34 mL cells
- Dionex vials for extraction collection (60 mL)
- Analytical balance

SOLVENT

Analytical grade n-hexane (or light petroleum ether especially composed of hydrocarbons with 6 carbon atoms)

ASE CONDITIONS

Temperature:	80 °C
Pressure:	6.67 MPa (1000 psi)
Time:	10 min
Static time:	10 min
Flush volume:	60%
Purge time:	60 s
Solvent:	n-hexane
Static cycle:	3

SAMPLE PREPARATION

The moisture content of all samples should be less than 10% (w/w). If greater than 10%, the moisture content should be reduced by drying the sample in an oven with a constant temperature of <80 °C. It is also important that the particle size of all samples be in the range of 2–5 mm. The samples should be ground using a mechanical mill without heating or changing moisture and oil content. It is essential that the oil extractions be carried out within 30 min of grinding, especially if the free fatty acid content of the extracted oil is to be determined.

PROCEDURE

Grind the sample into a particle size, specified above, using a mechanical grinder. Place a cellulose filter at the bottom of the extraction cell (11 mL or 34 mL cell). Fill the cell with 5–10 g of the sample. Put ASE Prep DE on top of the sample and leave the head space of the cell around 1/5 of the cell length. Place another cellulose disk on top of the ASE Prep DE and assemble the extraction cell. Place the extraction cell into ASE. Set the method conditions on the ASE system and start the extraction. Upon completion of

Table 1. British Standard ISO 659:1999

Sample size	10 g ground seeds
Solvent	n-hexane
Total volume of solvent used	~150 mL
Extraction 1	4 h, heat and grind
Extraction 2	2 h, heat and grind
Extraction 3	2 h
Total extraction time	10 h

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the extraction, transfer the extracts to a round-bottom flask and assemble the evaporation apparatus. Weigh the extracted oil and calculate the percentage of oil content using the following equation:

% Oil Content = Weight of Extracted Oil (g) / Weight of Sample (g) × 100

RESULTS AND DISCUSSIONS

Accuracy and Reproducibility

The extraction in Table 1 was conducted on Jatropha seeds using both the British standard (BS EN ISO 659:1999) and the ASE method. The percentage of oil in the Jatropha seeds was determined using a conventional Soxhlet extraction apparatus according to BS EN ISO 659:1999 and the result was 47.2 ± 0.7% weight oil / weight kernel (n = 4) with a total extraction time of 10 h. For ASE, the percentage of oil extracted was approximately 48.5 ± 1.3% weight oil / weight kernel (n = 3) when extracted at 80 °C and around 50.6 ± 0.5% weight oil / weight kernel (n= 3) when extracted at 100 °C. It should be noted that the sizes of sample portion for BS EN ISO 659:1999 and ASE were 10 g and 5 g, respectively. It can be concluded that the results obtained from the ASE method are consistent with those obtained from the standard method (Figure 1). In addition, the repeatability of the test results was very good. The ASE method can also be effectively used when the sample portion is only 3 g. The percentage of extracted oil is around 49.5 ± 2.6% weight oil / weight kernel (n =3). Although the standard deviation is slightly higher compared to the results obtained from BS EN ISO 659:1999 and ASE when using 5 g of sample, the results from 3 g are consistent with the 5 g results (Figure 2).

CONCLUSIONS

ASE is a very effective method for oil extraction in the determination of oil content in Jatropha seeds. The test results are very consistent with the results obtained from the British standard. The results obtained from the ASE method have high accuracy and precision. In addition, the ASE method can be applied in the determination of oil content when a limited amount of sample is available. It should also be emphasized that oil extraction using the ASE method requires less than 1 h compared to the 10 h consumed by Soxhlet extraction.

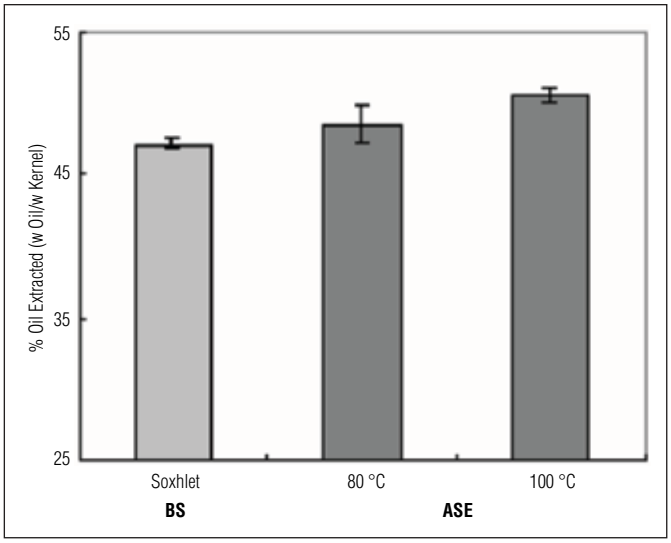

Figure 1. Percentage of oil extracted using the British standard and ASE method.

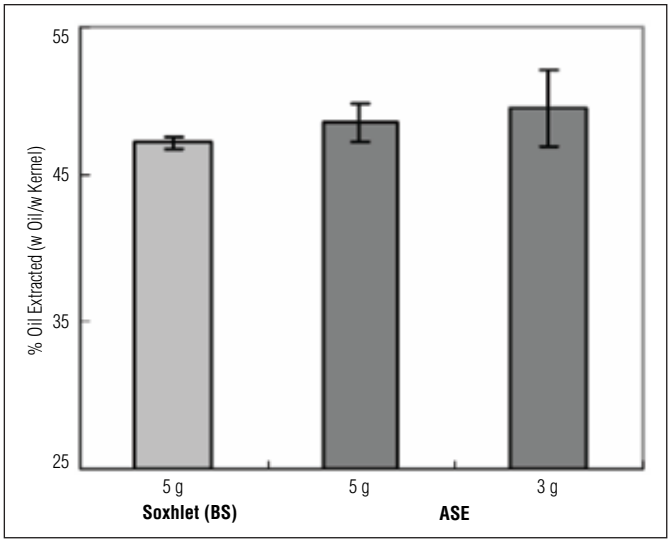

Figure 2. Percentage of oil extracted using different amounts of sample.

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