# BIO-DIESEL PRODUCTION FROM CAMELINA OIL, WASTE COOKING OIL AND TALLOW

**Authors**

---

B. Rice, A. Fröhlich and R. Leonard
**Crops Research Centre**
**Oak Park, Carlow**

Teagasc acknowledges with gratitude the support of the European Union Framework Programme in the financing of this research project

ISBN 1 901138 67 4

September 1998

Teagasc
The Science of Farming and Food

EUROPEAN UNION
European Agricultural Guidance and Guarantee Fund

---

Teagasc, 19 Sandymount Avenue, Dublin 4

## CONTENTS

## SUMMARY

The high cost and lack of availability of raw materials is limiting the expansion of bio-diesel production. The aim of this project was to examine the potential of alternative vegetable oils, oil wastes or animal fats as bio-diesel feedstocks, and the performance of road vehicles using bio-diesel blends made from these materials. Three feedstock materials were considered: waste cooking oil from the catering industry, Camelina oil, and beef tallow.

Thirty-four 300-litre pilot-scale batches of these materials were esterified, and yields and bio-diesel properties were measured. Five growers produced about 6 ha of camelina sativa on their set-aside land. Vehicle performance trials were carried out with five fuel blends involving bio-diesel and mineral fuel. A plant to produce approx 3000 tonnes per annum of bio-diesel was specified and costed.

The work has concluded that waste cooking oil is the most promising raw material for the immediate start-up of bio-diesel production. A proportion of camelina oil could also be used. Further work is required to overcome technical problems with tallow.

The cost of bio-diesel production in a 3000 t/yr plant from these raw materials was estimated at from 27 to 32 pence per litre of fuel. Reduction of excise on bio-diesel to the level applied to heating and agricultural fuels would make its final price competitive with mineral diesel for road use. The excise remission could be justified by a reduction of global warming and harmful vehicle exhaust emissions, and the provision of a safe disposal system for otherwise waste materials.

## INTRODUCTION

Preliminary studies of the potential for bio-diesel production in Ireland arrived at the conclusion that a small-scale bio-diesel plant based on rape-seed oil could not be viable because of high raw material cost. The following alternative raw materials were examined:

(i) *Waste cooking oil from the catering industry:* About 5000 t of this material is collected for recycling at present. It is thought that about 10,000 t of waste oil is available for collection, mainly from the catering trade.

(ii) *Oil from alternative oil-seed crops:* One alternative crop, *Camelina sativa,* is being examined at Oak Park. Its oil yield is similar to that of rape, but it requires lower fertiliser and pesticide inputs, which leads to a lower cost and a more favourable energy ratio.

(iii) *Tallow:* About 60,000 tonnes of beef tallow is produced annually in Ireland. Much of the lower-grade tallow has been used in animal feed compounds, but the future of this market has been brought into question by the outbreak of BSE in 1996. Legislation to ban the use in animal feeds of tallow from brain and spinal offals is to take effect throughout the EU in 1998, and will generate a supply of tallow for which there is no existing market.

## METHODS

Camelina seed, mainly from the variety Hoga was obtained from crops grown on farms in County Wicklow. The seed was pressed with a KOMET S-874 (IGB Montforts Gmbh) bench-scale press fitted with a seed hopper, oil tank and meal bagging arrangement to allow continuous operation. Suspended solids were removed from the oil by settling and decanting.

Oil for laboratory use was refined by the addition of sufficient potassium hydroxide in 70% aqueous solution at 30-35$^{o}$C, and the solids were removed by centrifuging. Waste cooking oil and tallow were obtained from Dublin Products, Dunlavin, Co. Wicklow.

Esterification was carried out using methanol and potassium hydroxide, and both the glycerol and water layers were allowed to separate overnight. Residual water was removed by heating. In some batches, methanol was removed by distillation before washing. Before esterification, waste cooking oil was heated to 40$^{o}$C, and tallow to 50$^{o}$C. Camelina oil was esterified at ambient temperature.

Over 7 tonnes of waste cooking oil was esterified in twenty-two 250-350 kg batches after preliminary cleaning by Dublin Products. Eight batches of camelina oil and four of beef tallow were esterified.

The effect of storage temperature on the properties of the tallow was examined. The cost of biodiesel production in a 3000t/yr plant was estimated.

# RESULTS AND DISCUSSION

## Waste cooking oil

The fatty acid composition of the esters varied considerably from batch to batch, though it appeared that in most cases rape-seed oil was the predominant component (Table 1). Other oil properties also varied considerably, with acid values from 1.9 to 7.4 and water contents from 1 to 5% (Table 2).

**Table 1:** Fatty acid composition of waste cooking oil and rape-seed oil esters

| Oil | Waste cooking | Rape-seed |
|---|---|---|
| C16:0 | 4.7 - 14.5 | 6.1 |
| C16:1 | 0 - 2.4 | |
| C18:0 | 1.4 - 5.9 | 2.3 |
| C18:1 | 52.9 - 65.8 | 56.0 |
| C18:2 | 15.4 - 20.7 | 24.2 |
| C18:3 | 4.1 - 8.8 | 6.5 |
| C20:0 | 0.6 - 1.0 | 0.3 |
| C20:1 | 0.4 - 2.3 | 0.3 |

**Table 2:** Properties of waste cooking oil and ester batches

| Batch no. | Range | Average | Specification |
|---|---|---|---|
| Acid value of oil | 1.9-7.4 | 5.3 | |
| Yield before washing ( %) | 63-87 | 78.3 | |
| Yield after drying (%) | 59-86 | 73.0 | |
| Methyl ester properties | | | |
| Acid value[i] | 0.02 - 0.73 | 0.28 | <0.5* |
| Iodine no.[i] | 99 - 118 | 105 | <115* |
| Free glycerol (%) | 0 - 0.02 | 0.0075 | <.03* |
| Total glycerol (%)[ii] | 0.10 - 0.57 | 0.24 | <0.25* |
| Methanol content (%) | 0 - 0.15 | 0.04 | <0.3* |
| Water content (%) | 0.07 - 0.19 | 0.11 | <.05* |
| Density (g/cm$^3$) | 0.877 - 0.888 | .883 | 0.86-0.9* |
| Kin. viscosity, 40$^o$C (cSt) | 4.6 - 5.1 | 4.86 | 3.5-5.0* |
| CFPP ($^0$C) | 0 to -14 | -5.3 | <0 summer*.<br><-15 winter* |
| Ash content (%) | 0 - 0.019 | 0.007 | 0.01* |
| CCR (%), 100% dist. res[iii] | 0.04 - 0.14 | 0.090 | 0.1** |

[i]Methods recommended in EU draft specification (Commission of European Communities, 1993)
[ii]Handbook of analytical methods for methyl esters used as diesel substitutes, FICHTE, Vienna.
[iii]Determined in the laboratories of Bundesanstalt für Landtechnik, Wieselburg, Austria
* Commission of European Communities (1993)
**O Norm C 1190 (1995)

Results show that it was possible to obtain high yields of methyl ester from refined waste cooking oil, though somewhat lower than from refined rape-seed oil (Table 3). Esterification of crude waste oil gave much lower yields, due to the high levels of free fatty acids in the oil.

**Table 3:** Yields of methyl ester from crude and fatty acid free waste cooking oil

| Method | FA free WCO (%) | Refined rapeseed oil (%) | Raw WCO (%) |
|---|---|---|---|
| Low KOH | 97.3 ± 1.2 | 98.5 ± 0.36 | 83.9 ± 0.94 |
| High KOH | 91.3 ± 0.5 | 92.6 ± 0.77 | 77.7 ± 0.7 |

Average density of the methyl ester was 0.883 $g/cm^3$, well within specification limits. Kinematic viscosity was from 7.1 to 8.8 cSt (average 7.7), also within specification. The high level of unsaturated fatty acids in the oil gave iodine values from 99 to 117, generally within specification.

Cold filter plug point (CFPP) was -5.3$^o$C on average, but varied over a wide range from 0$^o$C to -14$^o$C. One of the factors identified as contributing to this variation was the fatty acid composition of the ester. Higher proportions of the high-melting-point saturated fatty acids led to higher CFPP values.

On average, the occurrence of temperatures below -10$^o$C is less than one hour per year in Ireland. This suggests that a CFPP of -10$^o$C would be very safe, and that a value of -8$^o$C (exceeded only 4.8 hours per year) should very rarely cause problems. For summer use, a CFPP limit of 0$^o$C should be adequate.

The effect of CFPP-depressant additives on the low-temperature performance of waste cooking oil esters was inconsistent. Some samples showed improved CFPP levels, others gave lower pour points but unchanged CFPP.

An alternative approach is to blend the methyl ester with mineral diesel oil. Blends of equal proportions of waste cooking oil ester and mineral diesel gave CFPP values close to what would be needed in the Irish climate (Table 4).

**Table 4:** Low-temperature properties of blends of waste cooking oil ester and mineral diesel

| WCO ester % | Mineral diesel % | Cloud point $^oC$ | CFPP $^oC$ | Pour point $^oC$ |
|---|---|---|---|---|
| 100 | 0 | +3 | -3 | -3 |
| 80 | 20 | +3 | -5 | -6 |
| 60 | 40 | +3 | -8 | -6 |
| 40 | 60 | +3 | -11 | -9 |
| 20 | 80 | +3 | -12 | -18 |
| 0 | 100 | +3 | -15 | <-21 |

## Camelina oil

The ester yields for camelina oil were higher than for waste cooking oil. The fuel properties of the methyl ester were also within specifications with the exception of cold filter plug point (CFPP). The average CFPP for the six batches was similar to that of rape methyl ester made from local rape oil, which is to be expected considering that both contain about the same amount of saturated fatty acids.

The effect of two CFPP depressants, CP7134 and Lubrizol, on the low-temperature properties of Camelina ester was examined. Both improved the CFPP and pour point to a level that would make the fuel acceptable in Irish winter temperatures.

The low-temperature properties of blends of varying proportions of Camelina ester and mineral diesel are given in Table 5. These figures suggest that, as with waste cooking oil, a blend with roughly equal amounts of Camelina ester and mineral diesel would have adequate low-temperature properties for Irish winter conditions.

**Table 5:** Low-temperature properties of blends of Camelina ester and mineral diesel oil

| Camelina ester % | Mineral diesel % | Cloud point $^oC$ | CFPP $^oC$ | Pour point $^oC$ |
|---|---|---|---|---|
| 100 | 0 | +3 | -3 | -4 |
| 80 | 20 | +3 | -7 | -6 |
| 60 | 40 | +3 | -9 | -9 |
| 40 | 60 | +3 | -11 | -12 |
| 20 | 80 | +3 | -13 | <-18 |
| 0 | 100 | +3 | -15 | <-21 |

## Beef tallow

Beef tallow has a melting point of about 40°C. This raises a question as to whether it should be stored at temperatures above its melting point for convenient handling before processing, and whether there are any problems associated with this approach, in particular an increase in free fatty acids.

A trial was carried out in which No. 2 grade beef tallow (free fatty acid level less than 5%) was stored at three temperatures (20, 45 and 60°C) for a period of nine weeks, during which the free fatty acid levels were monitored. The trial results show that temperature has a pronounced effect on the rate of increase of free fatty acids (Fig. 1). If tallow has to be stored in liquid form, its temperature should be kept as close to its melting point as possible, and if it has to be stored for more than a month it should be stored in the solid state.

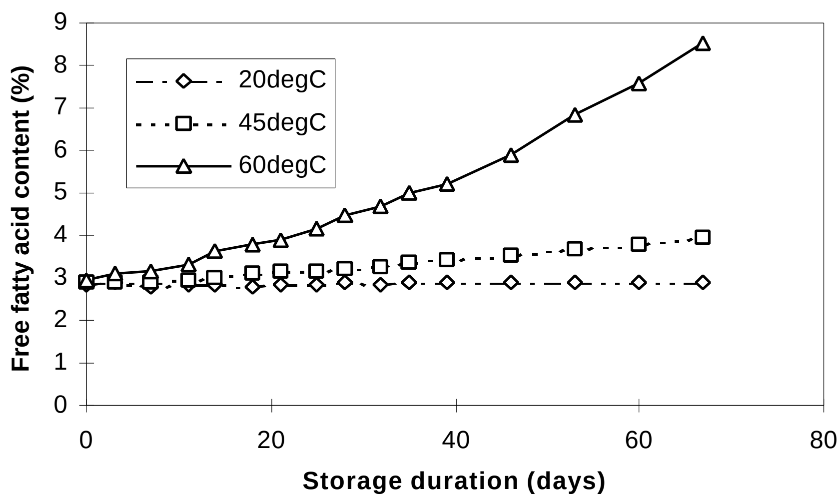


**Fig. 1:** Increase in free fatty acid content of beef tallow with storage duration and temperature

Four batches of No. 2 grade beef tallow (free fatty acid content less than 5%) were esterified. At the first esterification, after one week's storage at 65°C, the FFA content was 4.1%. A yield of 78% was obtained, and all the reaction-specific ester properties except total glycerol were within specification

A second batch was esterified after a further nine weeks, during which the tallow was stored at 70°C. By this stage the FFA content had increased to 20.1%. As the rise in FFA level had not been anticipated, insufficient KOH was used in the process. Esterification was not complete, the ester and glycerol did not separate cleanly, and levels of triglycerides and total glycerol were excessive. A second esterification improved the ester properties, but a very low yield was obtained. With subsequent batches, care was taken to control free fatty acid levels by minimising storage duration and temperature before esterification. Yields were somewhat improved, though they remained lower than with vegetable oils.

A problem with all the tallow batches was that layer separation was less well defined than with the vegetable oils, leading to higher ester losses during glycerol removal and washing. The problem appeared to be due mainly to the presence of suspended solids in the tallow, which accumulated at the interface during separation. Attempts to remove this material by filtration were only partially successful; at the high temperatures needed to ensure that the tallow remained liquid, much of the solids passed through the filter.

Of the fuel-specific properties, CFPP and viscosity values were not within specification. The iodine value was very low, due to the high level of saturated fatty acids. The CFPP of all the tallow ester batches was about 15°C. This is clearly unacceptable, even in the warmest climates.

To date, the effect of two CFPP-depressant additives on the properties of beef tallow ester have been examined. While both gave satisfactory results with vegetable oil esters, they had a negligible effect on tallow ester, even at high rates (Table 6).

**Table 6:** Effect of additives on the low-temperatures of beef tallow ester

| | Cloud point °C | CFPP °C | Pour point °C |
|---|---|---|---|
| Tallow ME no additive | +19 | +14 | +18 |
| Tallow ME + 5000 ppm CP7134[a] | +18 | +14 | +17 |
| Tallow ME + 5000 ppm Lubrizol 7670[b] | +18 | +14 | +15 |

a. Product of Elf Aquitaine SA. b. Product of Lubrizol Ltd, Merseyside, UK.

Blending of tallow ester with mineral diesel offers the only immediate possibility of achieving reasonable low-temperature properties with fuels containing tallow ester (Table 7). Blends with up to 20% tallow ester appear to be sufficiently tolerant of cold conditions for use in Ireland.

**Table 7:** Low-temperature properties of blends of tallow ester and mineral diesel oil

| Tallow ester % | Mineral diesel % | Cloud point °C | CFPP °C | Pour point °C |
|---|---|---|---|---|
| 100 | 0 | +19 | +14 | +18 |
| 40 | 60 | +6 | 0 | +3 |
| 30 | 70 | +5 | -3 | -6 |
| 20 | 80 | +1 | -8 | -6 |
| 10 | 90 | +1 | -13 | -15 |

## Costs

The cost of waste cooking oil after assembly and cleaning was assumed to be £220 per tonne, with an ester yield of 87% and a glycerol yield of 33%. On this basis, the total cost of bio-diesel produced from waste cooking oil as sole feedstock (excluding the cost of additives) is about 32 p/litre (Table 8).

The tallow costing assumed a feedstock price of £150 per tonne, a yield of 80%, and a glycerol yield of 35%. On this basis, bio-diesel produced from tallow in a 3000 t/yr plant would have a cost of about 27p/litre (Table 8).

These costs are much higher than the present cost of mineral diesel, and could not be viable without some form of subsidy. The preferred form of subsidy in EU countries to date has been the full or partial removal of excise from bio-fuels. If this were to be applied in Ireland, the comparable price of mineral diesel would be about 35 p/litre, slightly greater than the bio-diesel production cost. The excise remission could be justified by a reduction of global warming and harmful vehicle exhaust emissions, and the provision of a safe disposal system for organic waste materials.

**Table 8:** Total cost of bio-diesel produced from waste cooking oil or tallow

| Feedstock | | WCO | Tallow |
|---|---|---|---|
| Oil cost | £/t oil | 220.00 | 150.00 |
| Esterification yield | % | 87.00 | 80.00 |
| Nett oil cost | £/t ester | 252.87 | 187.50 |
| Oil cost less glycerol value | £/t (ester) | 226.47 | 161.10 |
| | *p/litre (ester)* | *19.97* | *14.21* |
| *Capital cost* | *p/litre (ester)* | *5.14* | *5.14* |
| *Operating cost* | *p/litre (ester)* | *7.32* | *7.32* |
| *Biodiesel cost* | *p/litre (ester)* | *32.43* | *26.67* |

## CONCLUSIONS

- The experimental work carried out in this project shows that bio-diesel of acceptable quality can be produced from a number of low-cost raw materials. However, complete removal of road excise would be required for the fuel to be competitive in price with mineral diesel.

- Good quality bio-diesel can be produced from waste cooking oil. A quality control system would be required at reception, to monitor water content, free fatty acid levels and polymer levels in incoming materials.

- Camelina methyl ester has properties similar to rape methyl ester with the exception of its high iodine value. A vehicle test in which high levels of lubricating oil dilution occurred gave no indication that the high iodine value had any adverse effect on lubrication properties. Camelina oil from Irish-grown seed would be cheaper and more attractive to the grower than rape-seed oil

- A study of Irish temperature data suggests that a fuel for year-round use in the Irish climate should have a CFPP value not higher than -8$^{o}$C. With vegetable oil esters, the additives tested could help to achieve these levels.

- Beef tallow ester has poor low-temperature properties. Blending with mineral diesel in the ratio 1:4 can achieve an acceptable CFPP. Careful filtration at ambient temperature is required to avoid fuel filter blockages.

## PUBLICATIONS


**Fröhlich, A. and Rice, B.** 1996. Bio-diesel from waste cooking oil. *Proceedings of the 2nd activity meeting of the International Energy Agency, Vienna,* November, 1995, 11-18.

**Fröhlich, A. and Rice, B.** 1996. The preparation and properties of bio-diesel grade methyl ester from Camelina sativa. *Proceedings of International Conference on Standardisation and Analyses of Bio-diesel. Vienna*, November 1995, 235-240.

**Rice, B., Fröhlich, A. and Korbitz, W.** 1997. Bio-diesel production based on waste cooking oil: promotion of the establishment of an industry in Ireland. Final Report, ALTENER Contract XVII/4.1030/AL//7/95/IRL, European Commission.

**Rice, B., Bulfin, M., Kent, T., Fröhlich, A. and Lenehan, J.J.** 1997. Potential for energy production from agricultural and forest biomass in Ireland. Teagasc, Oak Park.

**Rice, B., Fröhlich, A. and Leonard, R.** 1997. Industrial possibilities of bio-diesel based on used frying oil in Ireland. *In: Workshop "Valorisation of used frying oils and fats in the Benelux." Brussels,* Dec. 10, 1997, 1-5.