

Quality Assurance for Rape-seed Oil as Vehicle Fuel

Study Commissioned by



Contributors



Teagasc, Crops Research Centre, Oak Park, Carlow
(B. Rice, C. Fogarty)



UNIVERSITY of LIMERICK
OLLSCOIL LUIMNIGH

University of Limerick
(J.J. Leahy)



Carlow Institute of Technology
(B.O 'Rourke)

Executive summary

The use of rape-seed oil as a fuel for suitably modified diesel engines has been expanding in Germany and other countries, and two small units to extract and filter oil for this purpose have commenced operation in Ireland. This technology could contribute to the transport biofuel substitution by the EU Liquid Biofuels Directive. German research is showing that a high oil quality is essential for trouble-free engine use. In this work the quality of the oil being produced in Ireland was examined, and actions needed to assure adequate oil quality identified. Methods of checking oil quality on site and in laboratories in Ireland and Germany were also considered.

Of the many oil properties that have been listed as having a bearing on engine performance, two emerged as of prime importance:

- Suspended solids, an excess of which can lead to blocked filters and injectors
- Acid value, excessive levels of which may lead to corrosion and abrasion of engine parts as well as degraded lubricating oil if its level is too high.

To control these properties, the results showed that the first requirement is clean seed, which in turn requires effective weed control and good seed cleaning before pressing. The oil should be filtered as soon as possible after pressing, and then allowed to cool. It should be stored in cool conditions as near to airtight as possible. It is also important to ensure that no re-contamination occurs due to solids residues in pumps, pipelines or storage tanks. Also fresh oil must not be allowed to become contaminated with older oil whose acid value may already have increased. The press should be set and operated in a way that avoids excessive oil temperatures.

Suspended solids, acid value and the other process-dependent properties such as ash, phosphorus and water contents should all remain within acceptable limits if these recommendations are put into effect. A monitoring programme is needed at the press site for suspended solids, acid value and water content. The ASG Quick-test kit is adequate for these measurements, with occasional verification tests by an outside laboratory.

Many of the oil quality deficiencies that were measured in this work can be attributed to start-up problems with the plants involved, e.g. excessive delays between pressing and filtration, inadequate seed cleaning facilities, and lack of experience in the management of the plants. The specification of both of the Irish commercial plants is adequate for the production of high-quality oil, but careful operation and regular monitoring will be required for this to be achieved on a consistent basis.

Recommendations for Good Oil Producing Operation

- Ensure that the seed is dried to about 7% moisture content and free from all contaminants, especially weed seeds.
- Keep the period between oil extraction and filtration/cooling to a minimum, preferably less than a week.
- Set and operate press in a way that avoids excessive oil temperatures.
- Allow oil to cool after pressing
- Store oil in cool conditions as near to airtight as possible
- Ensure that no re-contamination occurs due to solids residues in pumps, pipelines or storage tanks
- Do not allow fresh oil to become contaminated with older oil whose acid value may already have increased

Recommendations for Oil Quality Assurance

To ensure that high-quality oil is produced consistently, the following weekly records should be made on site:

- Seed moisture and temperature entering the press
- Oil temperature leaving the press
- Total suspended solids, acid value and water content of oil samples removed at exit from the filter press and at the point of dispatch. The ASG Quick-test kit could be used for these tests.

Monthly measurements by the appropriate standard methods should be made at an external laboratory of the following:

- Total suspended solids
- Acid value
- Water content (these values for comparison with Quick-test values)
- Phosphorus content
- Ash content
- Oxidation stability

Samples for these analyses should be removed after the filter press and at the point of despatch. Specimen *Oil Quality Control sheets* are included in the report as Appendix A.

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1. Introduction and objectives

It is now well established that rape-seed oil can provide a source of renewable fuel for diesel engines. The main problem is a high viscosity and vaporisation temperature, which could lead to pumping, atomisation and combustion difficulties. These problems can be overcome in either of two ways: by further processing of the oil to improve its pumping and combustion properties (usually achieved by esterification and layer separation to produce biodiesel) or by some peripheral modifications to the engine to allow it to cope with the more viscous fuel. Engine conversion kits for this purpose are widely available. The second option has attractions in Ireland, at least in the short-term, for a number of reasons. Plants can be established quickly, and so could make an immediate if small contribution to the achievement of Ireland's substitution target in the Liquid Biofuels Directive (Commission of European Communities, 2003). The small operating scale of cold-pressing oil extraction plants could be achieved without undue difficulty, and the capital investment required to launch such a project would be relatively low. In the event of a biodiesel plant being established at some stage in the future, the option of sourcing some of the oil requirement from these extraction plants would still be available.

For a successful production and use of unprocessed rape-seed oil as fuel, recent German research is showing that the quality of the oil is very important. This is particularly the case when the oil is extracted in small cold-pressing plants, since there are no post-extraction processes other than filtration to improve its quality. Much information on the effect of seed quality, plant specification and plant operating parameters on oil quality is now being established for these plants. A voluntary trading standard for fuel-grade rape-seed oil (the RK Standard, Remmele et al; 2000) has been drawn up in Germany, based on considerable research and practical experience. This standard specifies limits for 15 oil properties and test procedures for their measurement. The oil properties that are affected by the seed quality and extraction/filtration process are also identified.

The test methods in the RK Standard are defined in EN, ISO or ASTM standards. Some are relatively simple, e.g. titrations, others would require complex or costly equipment or facilities and a high level of operator skill and would be very difficult to carry out at oil-press sites. For the key process-dependant parameters, where the standard test method is unsuitable for on-site application, simple tests to provide some on-site guidance to the plant manager are needed. For this purpose, one German company, ASG GmbH, is marketing a Quick-test kit for simple estimation of three critical properties – acid value, water content and suspended solids.

In comparison with the testing of mineral diesel fuel according to the requirements of EN 590 (European Committee for Standardisation, 1999), there are many differences in the properties to be measured and the methods used to measure them. Facilities for the testing of mineral diesel therefore have limited application to the testing of vegetable oil as fuel for diesel engines.

There are currently three oil press units producing oil for use as vehicle fuel in Ireland; an experimental-scale unit at Teagasc, Oak Park and two small commercial cold-pressing units (presses A and B). Several other units are at various stages of planning.

The two commercial units each have a screw press with a seed throughput of 100-150 kg/hour (Straehle 130), a high-capacity filter press to remove most of the suspended solids, and a final candle filter to complete the oil clarification process. Various heaters and/or heat-exchange units are used to raise the seed temperature before pressing and the oil temperature before filtration, and to cool the oil-seed cake prior to storage.

The Oak Park unit is a small 10 kg/hr screw press (IGB Monforts, S-87G); oil clarification is achieved by allowing the oil to cascade through a series of sedimentation tanks.

If vehicles are to run without problems on the fuel produced in these units, the quality of the oil must be assured. This requires that the key oil quality factors that are affected by the extraction/filtration process and that in turn affect engine performance need to be clearly identified, limits for the values of these properties need to be determined and simple methods of measuring or estimating these values need to be established.

The objectives of the present work are as follows:

- To identify the key process-dependant oil properties that need to be measured frequently and the facilities and equipment needed to make these measurements.
- To assess the possibility of making the tests on site.
- To make occasional measurements of the quality of the oil produced at the two commercial plants in operation.
- For properties whose rigorous measurement following the appropriate standard is not possible on site, to explore the possibility of substituting simple indicative or qualitative tests.
- Where the measured levels of key properties exceed acceptable limits at the two commercial plants, to establish the reasons for these problems so that corrective adjustments can be made to either the plant design or operating procedure.

Oil analyses were carried out at the following laboratories:

- Teagasc, Crops Research Centre, Oak Park, Carlow
- University of Limerick (UL), Plassy, Limerick
- Carlow Institute of Technology (CIT), Carlow
- ASG Analytic-Service GmbH, Trentinger Ring 30, D-86356 Neusass-taefertingen, Germany
- SGS Ireland Ltd, Lakedrive 3026, Citywest Business Campus, Naas Rd., Dublin 24.

2. Oil quality parameters, limits and measuring methods

2.1 The RK Standard

The RK quality standard is used in Germany as a trading standard for rape-seed oil as fuel (Table 1, Remmele et al., 2000). It lists 15 fuel parameters with corresponding testing methods and limiting values that on one hand characterise rape-seed oil and on the other hand lay down quality criteria for fuel use (Table 1). It is the only existing quality standard for unesterified fuel-grade vegetable oil.

As characteristics of rape-seed oil, the following properties are included in the standard: density, flash point, calorific value, kinematic viscosity, low temperature behaviour, cetane number, carbon residue, iodine number and sulphur content.

Variable properties that are affected by crop agronomic practices, seed drying and storage and oil extraction and storage should be subject to regular quality checks. These are identified as suspended solids, acid value, oxidation stability, phosphorous content, ash content and water content (Remmele et al., 2000). Viscosity could also increase after long-term storage in poor conditions. Although not included in the RK-standard, peroxide level could serve as an indicator of deterioration in storage.

2.2 Effects of process-dependent oil quality parameters on engine performance

2.2.1 Suspended solids (contamination):

The amount of suspended solids in the raw plant oil is very much affected by the properties of the oil seed and the oil pressing method. Levels of 5-10% are usually found in cold-pressed oil immediately after extraction, whereas values below 25 mg/kg are required by the RK Standard. Hence the design and operation of the clarification process has a very important role in achieving acceptable oil quality.

A high level of suspended solids leads to blocked fuel filters and injectors. It may also cause damage to the atomiser and the injector pump. Furthermore it may lead to deposits in the combustion chamber and an increase of harmful emissions (Thuncke and Kern, 2002).

2.2.2 Acid value:

The acid value is an indicator of the content of free fatty acids in plant oil. It is known to be affected by the duration and conditions of storage of the oil. High acid value in the fuel leads to corrosion, abrasion and deposits in the engine. Furthermore the free fatty acids may react with the alkaline components of the lubricating oil and affect its lubricity (Thuncke and Kern, 2002).

2.2.3 Oxidation stability:

Oxidation of oils and fats is facilitated by a supply of oxygen and by the presence of heat, light and metal catalysts (e.g. copper, iron). Oxidation and polymerisation processes may also lead to the formation of insoluble compounds, which may cause filter blockages. Harmful interactions between the fuel and the engine lubricating oil also become more likely (Remmele, 2002). Natural antioxidants in plant oil can control the oxidation process to a large extent (Thuncke and Kern, 2002).

2.2.4 Phosphorus content:

Phosphorus in plant oils is present as phospholipids. With an increasing amount of phospholipids the oxidation stability is reduced. Furthermore phospholipids tend to form particles with water and can cause filter blockage. Phosphorus reduces the combustion temperature, leads to deposits in the combustion chamber and eventually changes the emission behaviour. The life and efficiency of oxidation catalytic converters are very sensitive to the presence of phosphorus compounds. The phosphorus content of unrefined plant oil can be controlled by optimising the operating conditions of the press (Remmele, 2002).

2.2.5 Ash content:

The ash content describes the proportion of inorganic solids in the fuel. High ash contents can be caused by contamination of the seed or oil with dust (Thuneke and Kern, 2002). High ash content of fuels leads to abrasion of the injector pump and nozzles and the combustion chamber (Remmele, 2002).

2.2.6 Water content:

High water content leads to crystal growth at low temperatures and hence causes filter blockage. Because of the high pressures in modern injection systems, free water is released that could damage the injection system. At the boundary layer between water and fuel, the growth of micro-organisms is promoted which can block the fuel filter and promote the ageing process (Remmele, 2002). In plant oils the water content is influenced by seed moisture, refining process parameters, condensation effects and water uptake during storage (Thuneke and Kern, 2002).

Properties / Contents	Unit	Limiting Value		Testing Method
		min.	max.	
characteristic properties for Rapeseed Oil				
Density (15 °C)	kg/m ³	900	930	DIN EN ISO 3675 DIN EN ISO 12185
Flash Point by P.-M.	°C	220		DIN EN 22719
Calorific Value	kJ/kg	35000		DIN 51900-3
Kinematic Viscosity (40 °C)	mm ² /s		38	DIN EN ISO 3104
Low Temperature Behaviour				Rotational Viscometer (testing conditions will be developed)
Cetane Number				Testing method will be reviewed
Carbon Residue	Mass-%		0.40	DIN EN ISO 10370
Iodine Number	g/100 g	100	120	DIN 53241-1
Sulphur Content	mg/kg		20	ASTM D5453-93
variable properties				
Contamination	mg/kg		25	DIN EN 12662
Acid Value	mg KOH/g		2.0	DIN EN ISO 660
Oxidation Stability (110 °C)	h	5.0		ISO 6886
Phosphorus Content	mg/kg		15	ASTM D3231-99
Ash Content	Mass-%		0.01	DIN EN ISO 6245
Water Content	Mass-%		0.075	pr EN ISO 12937

Table 1: The RK Standard for rape-seed oil (Remmele et al., 2000).

2.2.7 Peroxide value:

Peroxide value is not included as a parameter in the RK standard. Nevertheless it can be a useful early indicator of oxidative deterioration and a decrease in the effectiveness of the oil's own antioxidants. During oxidative deterioration the peroxide value first rises and then falls after reaching a maximum value. This leads to difficulties with the interpretation of occasional measurements.

During storage, peroxides are generated by oxygen access. A rise of viscosity and gummy deposits can be the consequence. Furthermore there are indications that plant oils damaged by oxidation may lead to thickening of the lubricating oil and therefore to engine damage. The deterioration by oxidation is supported by oxygen access, light and high temperatures and by the presence of metal catalysts (Thuneke and Kern, 2002).

2.3 Effect of characteristic oil properties on engine performance

Density is an important physical property in the handling and storage of the oil as fuel. It may also affect the behaviour of the oil in blends with other fuels of different densities. Flash point is a measure of the risk of fuel combustion in storage. Rape-seed oil has a very high flash point, and problems would arise only if it were mixed with more volatile fuels. Calorific value is a measure of the fuel's energy content. Very little variation would be expected in any of these properties between rape-seed oil samples.

The kinematic viscosity is also mainly a function of the oil seed and is used like the density and the calorific value to distinguish rape-seed oil from other oils. The viscosity of the fuel affects the pumping characteristics and the atomisation of the fuel during injection (droplet spectrum, geometry of the injection stream). As a result of the more difficult flowing, pumping and atomising behaviour, high viscosity may lead to cold-start problems.

Viscosity depends very much on the temperature. By heating the oil to about 90°C the viscosity can be reduced to a level near that of diesel fuel. Due to the danger of increased polymerisation, heating should only be carried out immediately before combustion (Thuneke and Kern, 2002). An increase in viscosity may be an indicator of excessive oxidation or polymerisation of the oil.

In addition to increasing viscosity, low temperatures may also initiate freezing of the oil. The normal measures of the onset of freezing are cloud point, cold filter plug point and pour point. A test method for low-temperature behaviour has not yet been included in the RK standard. Since vehicle conversion kits normally include some fuel heating, small variations in low-temperature properties are unlikely to cause significant problems.

Cetane number is a measure of the combustibility and capacity for self-ignition of the fuel in the combustion chamber. A measuring method has not yet been defined in the RK standard. Little variation would be expected between rape-seed oil samples.

The Conradson carbon residue (CCR) of a sample is the carbonaceous residue formed after evaporation and pyrolysis. It consists of organic and inorganic compounds and its measurement gives information about the tendency to carbonisation at the injector nozzles and residue generation in the combustion chamber (Thuneke and Kern, 2002). High carbon residues are also linked to polymer formation in the fuel or in the lubricating oil.

The iodine value is an indicator of the amount of fatty acids with double bonds in the oil. A low iodine value indicates a high degree of saturation of the oil. This value gives information about the possible creation of deposits at the injector nozzles and in the combustion chamber, about the storage stability of the oil and its tendency to polymerise, and about the possibility of adverse effects on the lubricating oil. With rape-seed oil the iodine value varies between 100 and 120 g/100g, depending on the variety and growing location. At values above 120g/100g, it is suggested that carbonisation may increase (Thuncke and Kern, 2002).

The sulphur content in plant oils is generally very low, usually less than 20 mg/kg. Hence the emissions of sulphur dioxide and sulphates that are accumulated on particles are at a low level (Thuncke and Kern, 2002). The sulphur content may fluctuate depending on the amount of sulphur present in the soil or applied as fertiliser to the rape plant, but is likely to be much lower than standard grades of mineral diesel.

2.4 Standard measuring methods for process-dependent properties

2.4.1 Suspended solids (*EN 12662, National Standards Authority of Ireland, 1999*):

An oil sample and fine filter paper is desiccated and weighed. The oil is sucked through the filter, which is then washed with a solvent, dried, desiccated and weighed again.

2.4.2 Acid value (*ISO EN 660, International Organisation for Standardisation, 1996*):

The acid value is determined by a simple titration. The free fatty acids are neutralized with potassium hydroxide (KOH). When all fatty acids are neutralized, the indicator (phenolphthalein or Alkaliblu) colour changes from dark green to orange.

2.4.3 Oxidation stability (*ISO 6886, International Organisation for Standardisation, 1996*)

A stream of air is passed through the oil sample at a temperature of 110°C. The air with gaseous breakdown products of oxidation is passed through a container of distilled water whose conductivity is monitored. The stability period is defined as the time up to the point where the conductivity increases rapidly.

2.4.4 Phosphorus content (*ASTM D 3231-99, American Society for Testing Materials, 1999*):

Organic matter in the sample is decomposed by ignition in the presence of zinc oxide. The residue is dissolved in sulphuric acid and reacted with ammonium molybdate and hydrazine sulphate. The absorption of the Molybdenum Blue complex is proportional to the phosphorus concentration in the sample and is read at approximately 820 nm in a 5-cm cell.

2.4.5 Ash content (*EN 6245*):

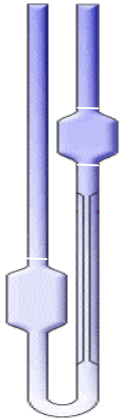
This is measured by burning the sample until only ash and carbon remain. The residue is ashed in a muffle furnace at 775°C and then weighed.

2.4.6 Water content (*ISO 12937, International Organisation for Standardisation, 2000*):

A weighed portion of oil is injected into the titration vessel of a Mettler Toledo DL31 Karl Fischer titrator. When all the water has been titrated, excess iodine is detected by an electrometric end point detector and the titration is terminated.

2.4.7 Peroxide number (Jacobs, 1958):

The determination of peroxide number is made by a titration in which 5 g of oil is dissolved in 30 ml of a mixture of 60% glacial acid and 40 % dichloromethane. Then 0.5 ml of saturated potassium iodide is added. The iodide reacts with the peroxides in acid solution. The I_3^- is then titrated with sodium thiosulfate. By adding starch to the solution a colour change is visible, when the I_3^- is converted into I^- . The peroxide number can be calculated from the consumed sodium thiosulfate.



2.4.8 Kinematic viscosity (ISO 3104, International Organisation for Standardisation, 1976):

Viscosity is measured with a U-tube viscometer. The viscometer has a defined capillary through which the oil flows. The tube is first filled up to a graduation mark over the left storage bulb (Fig. 1). The oil is then pressed or sucked up to fill the higher storage bulb in the right leg. This bulb has graduation marks above and below, and from the time needed for the oil to flow under gravity from the upper marker to the lower the kinematic viscosity can be calculated. A thermostatically controlled water bath is required to maintain the oil at 40°C for the duration of the test.

Fig. 1: U-tube viscometer.

2.5 Measuring methods for characteristic oil properties

For density measurement, the oil is filled into a flask with an exact known volume and a very narrow filling aperture. The oil is brought to a temperature of 15°C. The flask is weighed before and after filling with oil. From the difference of weight and the volume of the flask, the density at this temperature can be easily calculated. Flash point is measured with the Pensky-Martens apparatus. An oil sample is heated slowly and exposed to a flame at fixed temperature intervals. The flash point is the lowest temperature at which the flame causes the sample to burn. Calorific value is measured in a bomb calorimeter. A fuel sample is burned in an enclosed chamber, and the temperature rise in a surrounding water bath is measured. To measure carbon residue, a weighed sample is placed in a crucible and subjected to destructive distillation. The residue undergoes cracking and coking reactions during a period of severe heating. At the end of the heating period the test crucible containing the carbonaceous residue is cooled in a dessicator and weighed. Testing methods for low temperature behaviour and cetane number are not yet specified in the RK standard.

2.6 The ASG Quick-test kit

The quick-test kit is available from the company ASG Analytik-Service Gesellschaft, Trentiner Ring 30, 86356 Neuass-Täfertingen, Germany. It includes test methods for suspended solids, acid value and water content. The current price is €270 for a kit to test 25 samples. The methods for acid value and water content were developed by ASG; the method for suspended solids was developed by the Bayerischen Landesanstalt für Landtechnik (Remmele et al., 2000).

2.6.1 Quick determination of suspended solids:

The ASG Quick test for suspended solids is an attempt to realise the measuring method in DIN EN 12662 with a lower sample volume and without expensive weighing equipment.

A syringe is filled with 10 ml of rape-seed oil. Then the oil is pressed through a cellulose acetate filter membrane (0.5 μm) with the help of a syringe filter attachment. This process is repeated three times to achieve a total sample volume of 30 ml. The syringe is then used to press air through the membrane to force out most of the oil. Petrol ether (20 ml) is then filled into a new syringe and pressed through the membrane, to dissolve and wash any remaining oil from the membrane and filter housing.

The filter membrane is then removed and stored on a solvent-resistant surface while the solvent evaporates. The residue on the membrane filter is then visually assessed using a supplied chart on which filter membranes with different contamination levels are displayed (Fig. 2).

2.6.2 Quick determination of acid value:

This is a simplified method of carrying out the titration in ISO 660. A small flask is supplied containing 10ml of a mixture of sodium hydroxide, a solvent mixture and an indicator. The oil is dropped into the flask with the help of a syringe. If the colour change happens before a consumption of 4 ml oil, the RK-quality limit is exceeded. The colour change is visible in Fig. 3. From the consumption of oil before colour change, the acid value level can be estimated (Remmele et al; 2000).

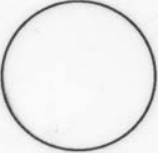














Übliche Filterbeaufschlagung im Schnelltest			Gesamtverschmutzung (DIN EN 12662) (mg/kg)
			9
			24
<hr/>			Grenzwert: 25 mg/kg
			34
			37
			51

Fig. 2: Comparator table for use in the ASG Quick test for suspended solids.

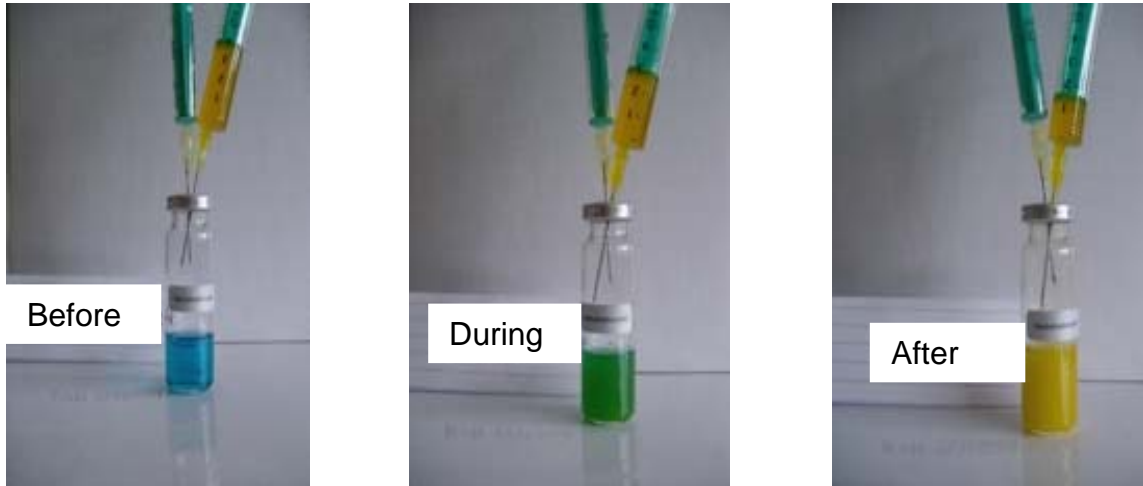


Fig. 3: Quick test for acid value, showing the indicator colours before, during and after the titration reaction.



2.6.3 Quick determination of water content

The ASG Quick test for determining the water content of rape-seed oil is similar to the quick test for the acid value. A flask containing Karl-Fischer reagent is supplied, whose volume is such that a colour change should occur when 2 ml of rape-seed oil at 0.075 mass-% water content is added. A syringe is used to drop the rape-seed oil into the flask while shaking vigorously. The water content can be estimated from the amount of oil added at colour change, the water content may be estimated (Remmele et al., 2000).

Fig. 4: Quick test for water content right before, left after colour change.

2.7 Oil quality from cold-pressing plants

The quality of rape-seed oil is of crucial importance if it is to be used without problems in diesel engines. Recent research by Remmele and Stotz (2003) is showing that cold-pressing extraction units in Germany are having difficulty in consistently producing oil to the RK-standard. Only four of thirty oil mills surveyed were consistently meeting the standard.

Values of density, iodine value, calorific value, flash point and kinematic viscosity were within standard and showed the least variations. Oxidation stability, carbon residue, phosphorus content, ash content and water content were less consistent but mainly within the limits of the RK-standard. Suspended solids and acid value were the main problems and were frequently outside the prescribed limits (Thuncke and Kern, 2002).

The importance of achieving the limit values of the RK-standard for the oil operating diesel engines is being shown by a German 100-Traktor-Demonstration Project where 100 tractors were modified to run on rape-seed oil (Schümann et al., 2003). Although the trial is not yet complete, in interim reports several operation failures are being attributed to inadequate oil quality

Widmann (1994) researched the influence of seed and press parameters on oil quality. As seed treatment measures, seed moisture content, seed temperature entering the press, and various thermal treatments before pressing were examined. Seed moisture content was the only factor to have a significant influence on oil quality. Excessive moisture leads to reduced extraction efficiency, problems with seed cake storage and high oil moisture contents. Very low moistures may reduce throughput and lead to high oil phosphorus contents. Moisture contents of 6.5 to 7.5% gives the best compromise between these various considerations.

Of the press parameters, press head temperatures between 60 and 80°C gave the best compromise. The phosphorous content was reduced at lower temperatures but the contamination with suspended solids rose. The optimum speed rotation was between 20 and 50 rpm.

All these tests were carried out with a Komet two-rotor DD 85 G press made by IBG Monforts but the relationships should also be valid for different types of presses from different manufacturers.

3. Work programme

3.1 Oil sample analyses

A total of 17 oil samples were taken from the three oil presses. Background data on seed quality, press settings, filtration system and oil storage was recorded at each sampling. Oil sampling was carried out to include as many variations as possible in operating conditions. The results of these analyses were compared with the RK standard limits.

Benchmarking tests were carried out at the ASG laboratories for the full list of properties, and at the mineral fuel laboratory of SGS Oil, Gas & Chemicals, Dublin for carbon residues. All 15 properties in the RK-standard were measured on one sample at the ASG laboratory. Other crop-dependent properties were measured at Teagasc, University of Limerick and Carlow Institute of Technology as indicated in Table 2 (Chapter 4). For these parameters, the measurements were made mainly to ensure that values for Irish crops were similar to those reported in overseas literature, and to identify laboratories with the facilities and expertise to make the measurements.

Where possible, the process-sensitive properties such as suspended solids, water content, acid value, phosphorus content and ash content were measured at more than one laboratory. Suspended solids and acid values were measured at all three laboratories. Water and ash contents were measured at Carlow Institute of Technology and University of Limerick. Phosphorus content was measured at Teagasc. Quick-test analyses of suspended solids, water content and acid value were made by Teagasc.

The feasibility of carrying out these tests on site (i.e. resources, time and skill needed) was also assessed. All results are reported in Chapter 4.

3.2 Oil quality investigations

In preliminary analyses of oil samples, a number of problems quickly became apparent:

Some of the acid values of the oil from press A were higher than expected, and exceeded the RK limit. A possible source of the problem was identified as weed seeds mixed with the rape seed. To examine this problem further, oil samples were analysed firstly from the same seed extracted in the Teagasc press, and then from a cleaned seed sample, again in the Teagasc press. Results are reported in Chapter 4.

The first samples from press B also had high acid values. In this case the problem appeared to be linked to the storage conditions of the oil between pressing and filtering. To further examine this problem, an oil storage trial was conducted at Teagasc, in which oil samples were stored in various conditions of temperature, stirring and exposure to air, and the rate of deterioration of the oil was measured. The details of this work are reported in Chapter 5.

The standard method of measuring suspended solids required very accurate weighing equipment; the filtration of large samples was slow and big differences were recorded in the results from different laboratories. It was therefore decided to investigate the use of a centrifuge to clarify the oil and an estimation of the solids content from the height of the sedimentation layer. Details of this work are reported in Chapter 4.

4. Results and discussion

4.1 Measurements of process-dependent parameters

Table 2 shows the results of analyses of 17 oil samples. Some comments on these results are as follows:

4.1.1 Suspended solids:

At the commencement of testing, neither of the commercial plants had fully commissioned their filter presses. As a result most of the early samples analysed (e.g. samples 1,3,4,12, Table 2, Fig. 1) had been clarified only by a period of sedimentation and did not pass through a final filter. It was therefore to be expected that these samples had high solids levels. Samples 2,5,10 and 13 passed through the filter press and final candle filter; they were much nearer to the required level of 25 mg/kg, and in most cases below it. The results show that great care is needed to attain the specified level consistently. In addition to the required filtration, regular cleaning of post-filtration storage tanks and handling systems is needed to avoid contamination of clean oil.

In samples with high suspended solids, difficulty was encountered in getting consistent results between testing laboratories (Table 2, Fig. 5). Great care was required in the mixing of samples before splitting, to ensure the solids were dispersed uniformly. For samples with high solids levels, the filter became clogged quickly and oil throughput was slow. Samples with low solids were easier to analyse and gave better agreement between laboratories.

The ASG Quick test for suspended solids was also difficult to carry out where solids levels were high. The pressure required to force the oil through the filter paper increased rapidly as the paper became contaminated. The test was much easier to carry out on clean samples, but estimation of the solids content by comparison of the contaminated filters with those on the supplied chart entailed some degree of guesswork. It was also limited by the fact that the highest level of suspended solids on the comparator chart was 51 mg/kg, so there was no guidance for estimates above this level. The method would give a reasonably clear indication where the RK limit was being exceeded, but it could hardly be considered reliable enough to guarantee that samples were within the RK limit, nor could it give much indication of the extent to which the limit was being exceeded.

No.	Test lab	Suspended solids (mg/kg)	Acid value (mg KOH/mg)	Oxidation stability (h)	Phosphorus content (mg/kg)	Ash content (mass %)	Water content (mass %)
1	CIT	189	3.53			0.012	
	Teagasc	243	3.42		9.3		
	Quick Test	>51	3.56				0.180
	ASG	114	3.35	1.3	12.0		0.253
2	CIT	25	2.88			0.009	0.261
	Teagasc	27	2.66				
	Quick test	10 - 20	2.58				0.067
	ASG	17	2.62		14.1		0.065
3	CIT	154	1.97			0.014	0.241
	Teagasc		1.81				

	UL	179	2.36			0.016	0.040
	Quick Test		2.50				0.075
4	CIT	178	2.51			0.011	0.091
	Teagasc		2.63				
	UL	217	2.53				0.070
	Quick test		2.50				0.084
	ASG				3.2		
5	CIT	61	2.11			0.080	0.076
	Teagasc	51	2.10				
6	Teagasc	23	2.67		4.1		
	Quick test	51	2.58				0.079
	ASG				10.0		
7	Teagasc		0.90				
	Quick test		0.95				
	ASG				7.0		
8	Teagasc	30	2.58		12.0		
	Quick test		2.67				0.054
9	Teagasc		2.93				
	Quick Test		3.08				0.047
10	CIT	23	2.32				0.070
	Teagasc	17	2.29		35.0		
	Quick test		2.50				0.068
	ASG	60	2.29	7.0	20.0	0.016	0.068
11			2.17				0.069
	CIT						
	Teagasc		2.11		29.3		
	ASG	282	2.08		20.9	0.006	0.060
	Quick test		2.11				0.068
12	Teagasc	252	0.52				
13	Teagasc	13	0.43		7.0		
	ASG	14		4.6	3.9		
14	Teagasc		2.12		15.4		
	Quick test		2.35				0.058
	ASG				11.6		
15	Teagasc		0.93		7.0		
	Quick test		0.95				0.056
	ASG				5.6		
16	Teagasc		1.82				
	Quick test		1.87				0.053
17	Teagasc		2.84				
	Quick test		2.85				0.055

Table 2: Analyses of process-dependent properties of rape-seed oil.

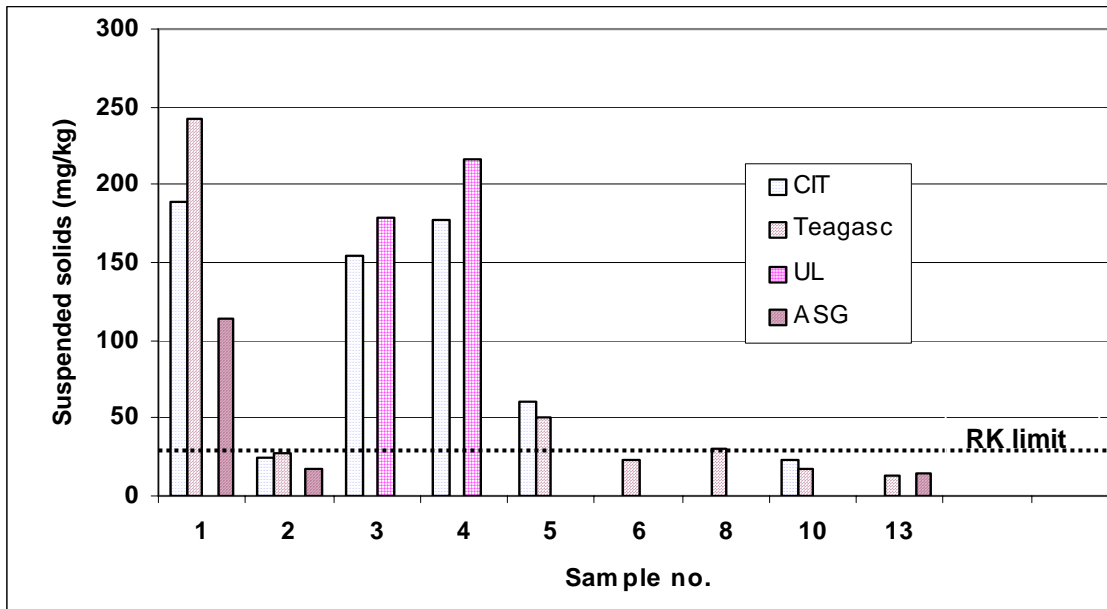


Fig. 5: Suspended solids measurements on nine oil samples.

A series of experiments were undertaken at University of Limerick to investigate if it was possible to determine the extent of oil sample contamination by centrifugation. Initially three samples whose contamination had been measured by the standard method (B.S. EN 12662, 1999) were tested. The solids levels measured in these samples were 18, 56 and 258 mg/kg respectively. Portions (1.0 mL) of oil were centrifuged in 1.5 mL conical centrifuge tubes in a Hettich Mikro 20 centrifuge. Two speeds were used, 5000 rpm and 10,000 rpm. The oil samples separated into a solid precipitate and very clear liquid.

After centrifuging the tubes were examined to determine if it was possible to quantify the amount of precipitated solids. As the quantity of precipitate was too small to measure visually, a bifocal light microscope with a graticule was used. It was possible to detect differences in heights of precipitate in the tubes between the low and high and medium and high samples but not between the low and medium samples. Precipitate heights were 3.5×10^{-4} cm for the low and medium samples and 3.2×10^{-3} cm for the highly contaminated sample. The results recorded were independent of centrifuge velocity for the two speeds tested. Measurements were complicated by the fact that the precipitate was not distributed homogeneously on the tube walls.

To examine the possibility of using larger sample sizes, 20-mL quantities of the oil samples were centrifuged using 30 mL Sarstedt tubes with conical ends in a ALC PK 110 Centrifuge at 5000 rpm. The oils tested included the original three as well as three dilutions of the high sample i.e. 0.5, 0.25 and 0.1. Flora sunflower oil was used as the diluent and three replicates of each sample were tested. The results in all cases proved inconclusive. It was not possible to accurately measure the volume of precipitate for two reasons

- Sample size was still too small
- The precipitate adhered to the wall of the conical portion of the Sarstedt tube and the manner of the distribution on the wall differed between samples which made it impossible to accurately determine volume.

From this experience, it seems unlikely that the centrifuge can be used as the basis for a satisfactory method of suspended solids measurement.

4.1.2 Acid value:

Initial acid value measurements from both of the commercial plants exceeded the RK limit of 2 mg KOH/g. In Press B (Samples 1-5, Table 2, Fig. 6), because of delays with the installation of the filter press, the oil had been stored for up to three months before sampling. In that period the oil was stirred occasionally and its temperature fell slowly from about 40°C down to ambient temperature. Oil samples removed from press B immediately after pressing gave an acceptable acid value of 0.9 (Sample 7, Fig. 7).

The oil deterioration in this case was rapid, and would suggest that oil stockpiling for even a few months might be a problem. This would make it very difficult to manage the extraction, storage and utilisation of the oil. For this reason it was decided to carry out a trial to explore further the effect of storage conditions on oil quality deterioration. The details of this trial are described in Chapter 5.

The first sample from press A also showed high acid values (Fig. 6), even though in this case there was no delay in filtration and the oil was freshly pressed. A second sample produced with no oil heating before filter pressing gave only slightly lower result (Fig. 6). The seed being pressed at this time was contaminated with weed seeds, mainly those of common dock (known to contain oxalic acid) and cleaver.

To establish whether this seed contamination was the cause of the high acid values, first a sample of the contaminated seed was pressed in the Oak Park press. This gave similar acid values to the oil from press A (Fig. 6). The seed was then passed through a seed cleaner. This removed some but not all the weed seeds, which were similar in size and other physical properties to the rape seed. This screening reduced the acid value well below the limit (Fig. 6). Clearly the weed seed contamination was affecting the acid value. If all the weed seeds could have been removed, a further reduction of acid value to a normal value for fresh oil might have been expected.

The three laboratories that measured acid value using the standard method (Teagasc, University of Limerick and Carlow Institute of Technology) got very similar results for the same samples, and these were also in good agreement with results from ASG. The test was easy to carry out in a normal laboratory. The accuracy of the acid value Quick test was also acceptable, and could be carried out at an oil-press site. On 11 samples where the Teagasc RK and Quick-test results could be compared, the mean acid values were 2.31 for the Quick-test and 2.32 for the tests done according to the RK standard (Fig. 7).

4.1.3 Oxidation stability:

This test requires equipment which was not available in any of the Irish laboratories. Three samples were analysed at the ASG laboratory. Two were below the 6-hour minimum (1.3 and 4.6) of the RK standard, one gave a value of 7 hours. The below-limit samples were both of oils that had been in storage with occasional mixing for several months, and also displayed excessive acid values. The fresh oil of Sample no. 10 exceeded the minimum required level, in spite of any adverse effect of the weed seed contamination described earlier.

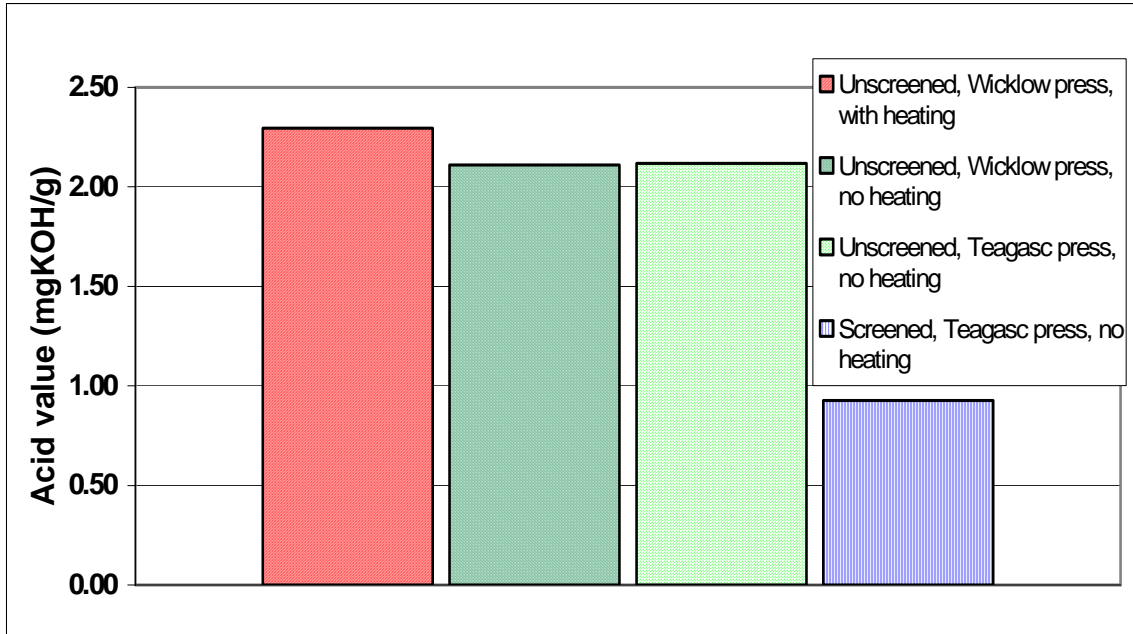


Fig. 6: Acid values of samples before and after weed seed removal.

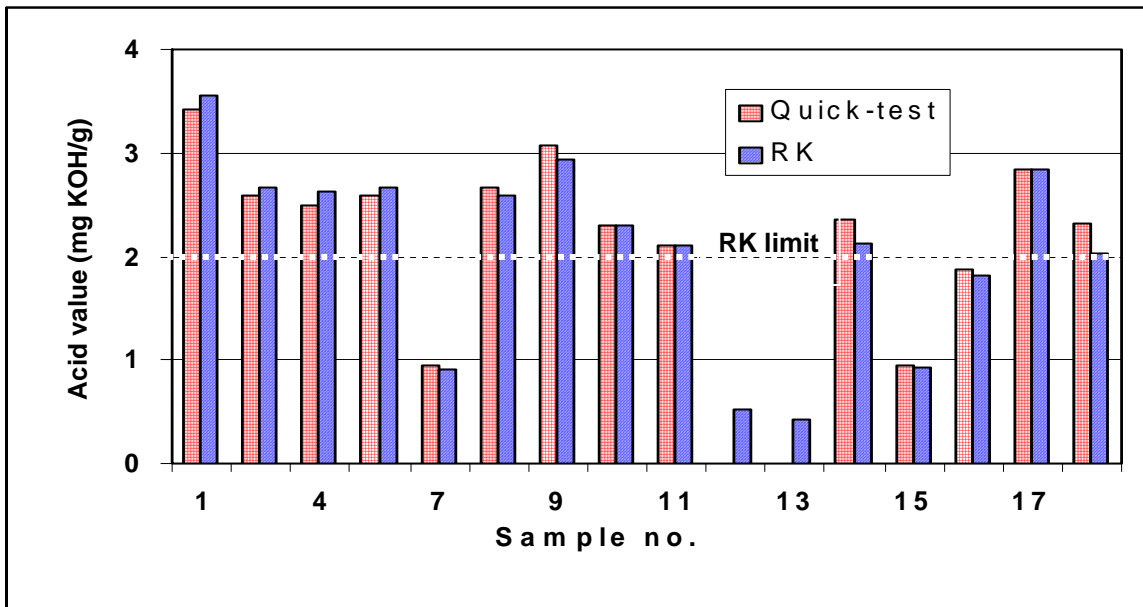


Fig. 7: RK and Quick-test acid value measurements on 11 oil samples.

4.1.4 Phosphorus content:

Of 11 samples analysed, nine gave P values below the RK limit of 15 mg/kg (Table 2, Fig. 8). The two out-of-limit samples (nos. 10 and 11) were those with a high level of weed seed contamination. The much lower P level in the screened sample of the same seed (no. 15) suggests that the weed seed contamination was the main cause of the excessive levels in samples 10 and 11. Overall the results suggest that the attainment of P levels within the RK limit should not be a problem as long as the seed is well cleaned and the press set to avoid excessive oil temperatures.

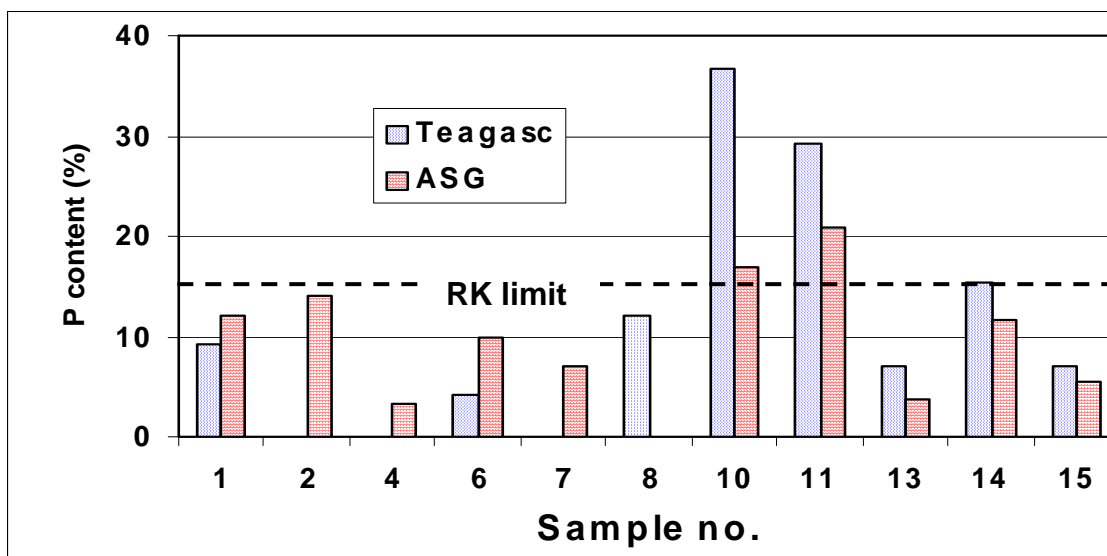


Fig. 8 : Phosphorus contents of 11 oil samples.

For the seven oil samples where the Teagasc and ASG results could be compared, agreement was not good. The accurate measurement of phosphorus is difficult. There are many sources of contamination, which can affect the outcome of the experiment. For example soaps and detergents contain phospholipids and also use surfactants that contain phospholipids. This means that both the glassware and the towels used to clean the glassware would be contaminated with phosphorus. For this reason all glassware must first be cleaned with acid (an operator hazard) and then rinsed with distilled water. Other reasons why phosphorus testing is unsuitable as an on-site test are the need for a spectrophotometer and fume cupboard and the amount of time required for one person to complete a set of tests (1 ½ days).

4.1.5 Ash content:

Almost half of the samples tested had ash contents over the RK limit of 0.01% (Table 2, Fig. 9). This is probably due to the high suspended solids levels in a number of incompletely-filtered samples. The effect of filtration on ash content is demonstrated by comparison of samples 1 and 2 (incompletely filtered and fully filtered samples of the same oil), and by samples 4 and 5 (also incompletely and fully filtered samples of the same oil). In both cases filtration reduced both suspended solids and ash content, in the first example to within the RK limit. While other factors play a role, it is likely that measures taken to control suspended solids (e.g. better filtration, cleaning of oil containers and handling equipment) and acid value (e.g. better seed cleaning) would go a long way to bring ash contents within the RK limit.

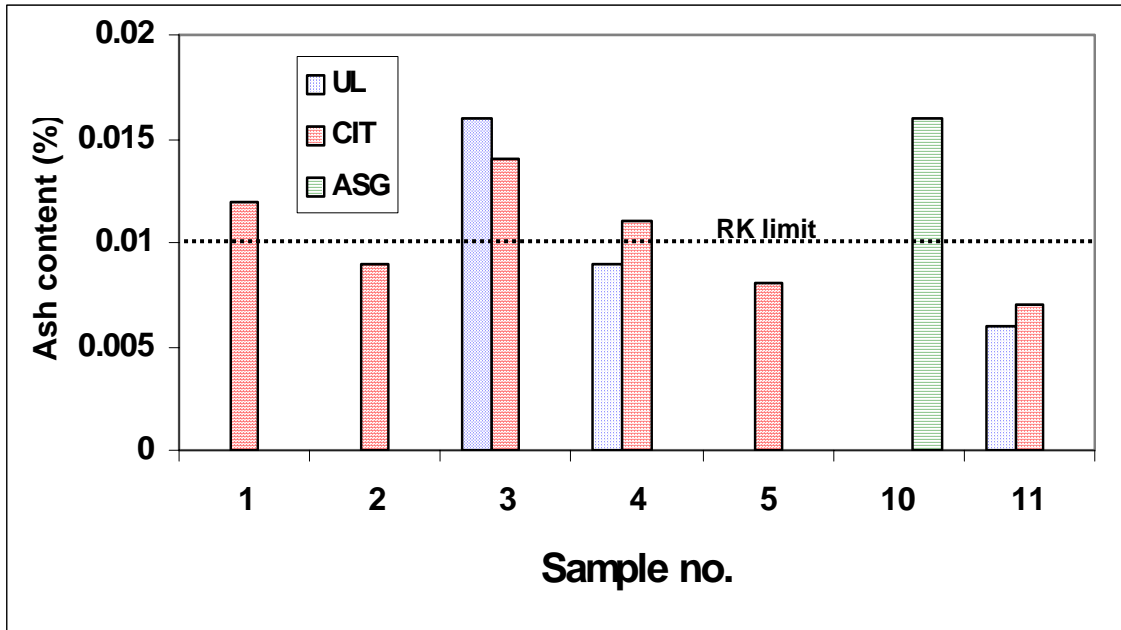


Fig. 9: Ash contents of seven oil samples.

4.1.6 Water content:

Of fourteen samples on which water content was measured, eleven were within the RK limit of 0.075%. Two of the over-limit samples (Sample 1 and 4, Fig. 10) were incompletely filtered and had high levels of suspended solids. When the oil was passed through a final candle filter, in addition to reducing the suspended solids the water contents were also reduced to within the RK limit (Samples 2 and 5 respectively, Fig. 10). For the within-limit samples, there was good agreement between the RK tests and the quick tests (Fig. 10). The lack of agreement between the two methods for Sample 1 with a very high water content may be due to a non-homogeneous distribution of water within the sample and insufficient mixing before analysis.

These results suggest that water content should not be a problem for oil that is properly filtered and stored. While the RK test requires expensive equipment that is not readily available, the ASG quick test provides a simple method that could be carried out at any press site.

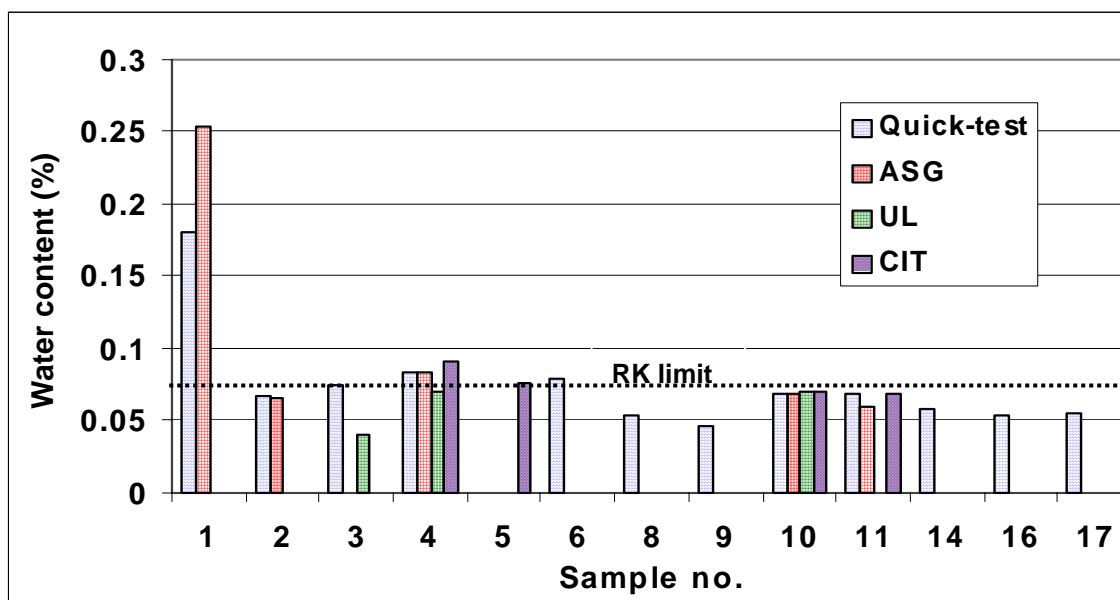


Fig. 10: Water content of fourteen oil samples.

4.2 Measurement of characteristic properties

Oil density in four samples was from 915 to 930 mg/kg, all within the RK range (Table 3). Agreement was good between the testing laboratories. Density could easily be measured at press sites, but would hardly be necessary unless some change in the oil source were envisaged.

Flash point was measured on only one sample sent to the ASG laboratory (Sample 10). At 248°C, it well exceeded the RK limit. Flash point measurement requires specialist equipment; even laboratories equipped for mineral diesel analysis may not be able to measure such a high flash point. Given that the flash point of pure rape-seed oil is very unlikely to fall below 220°C, there is little need for continuous monitoring. Flash point would only become an issue if rape-seed oil were mixed (intentionally or accidentally) with a more volatile fuel before storage. If this led to a significant reduction in flash point, more stringent safety precautions might then be required for the stored fuel.

Calorific value of seven samples varied from 38.3 to 42.0 MJ/kg, all well above the RK limit of 35 MJ/kg. The highly specialised measuring equipment is available at UL, but the need for such measurement would arise only if some other oil source were being examined.

Sample No.	Tested by:	Density (kg/m ³)	Flash (°C)	pt	Calorific value (kJ/kg)	Kinematic viscosity (mm ² /s)	Carbon residue (mass %)	Iodine value (g/100g)
1	IT Carlow	925						
	Teagasc	923				34.6	0.34	
	ASG						0.34	
	SGS						0.37	
2	IT Carlow	927						116
	Teagasc	919				34.6		
	ASG							119
3	UL				41.06	37.3		119
4	Teagasc	915						
	UL				41.03	37.5		123
8	UL				42.05			119
9	Teagasc						0.28	
10	Teagasc	919				34.2		
	ASG	920	248		36.48	34.1	0.34	118
	SGS						0.39	
11	UL				41.60	37.5		122
12	Teagasc					34.4		
13	Teagasc					34.4		
	ASG						0.32	
	SGS						0.28	
14	UL				41.30	37.8		122

Table 3: Analyses of characteristic properties of rape-seed oil.

The kinematic viscosities of ten samples all lay between 34 and 35 mm²/s, with very little variation and well below the RK upper limit of 38 mm²/s. Viscosity is easily measured with moderately priced equipment. However, viscosity measurement would be required only if a new oil source were being considered or as an indicator of severe deterioration of the oil.

The Cetane number of one sample (48.5) was measured by the ASG laboratory. No limit has been set for this property in the RK standard, and much research will be required to establish a basis for such a limit. In the interim, practical experience with vehicles running on the fuel suggest that its combustion properties are satisfactory. Carbon residue measurements of eight samples gave values from 0.28 to 0.39%, all below the RK upper limit of 0.40%. Results from Teagasc, SGS & ASG were in good agreement with each other. This measurement would be required only if the oil had been subjected to some treatment such as prolonged heating and/or exposure to air that might have accelerated deterioration.

Carbon residue measurement requires the same specialist equipment as is used with mineral fuel testing, but some adaptation is needed to achieve the desired pre-ignition time with a fuel of very low volatility.

5. Investigation of effects of storage conditions on oil deterioration

5.1 Introduction

The objective of the experiment was to explain the high acid values measured in oil samples from press B, and to set out management guidelines to ensure that within-limit acid values are achieved consistently.

The oil from press B had been extracted approximately four months before being analysed. It was stirred intermittently for three months in a double walled 5000-litre steel container before being pumped into 1000-litre containers where it was allowed sediment for one month. After this period the oil was pumped through an AMA candle filter for the final filtration. When analysed the oil had an acid value in excess of the RK limit. For this reason it was decided to store a number of oil samples in a range of conditions which might have occurred during the storage of the press B oil, and to monitor their deterioration. As indicators of oil quality, kinematic viscosity and peroxide number were monitored as well as acid value.

5.2 Experimental set-up

The oil for the trial was pressed at Oak Park ten days before starting the experiment. It was then allowed to settle for eight days. Samples with a high suspended solids content were removed at this stage. The remaining oil was filtered, and samples with low suspended solids were then removed. The actual suspended solids levels of the two sets of samples were 250 and 13 mg/kg, respectively.

Filtered and settled oil samples were stored in the following conditions:

- 50°C, open containers, stirred.
- 50°C, airtight containers, no stirring.
- Room temperature, open containers, stirred.
- Room temperature, airtight containers, no stirring.

The stirred samples were stored in 2L beakers that contained about 1.25L of oil and were covered lightly with aluminium foil. A 45 mm magnetic stirrer 45mm in length was placed in each container. The unstirred samples were kept in airtight 1-L polyethylene bottles completely filled with oil. All samples were stored in darkness.

Acid value and kinematic viscosity were determined using the RK standards. The peroxide number was determined by the procedure set out in Jacobs (1958). Over a period of 72 days nine sets of samples were taken at roughly equal time intervals and measurements to determine acid value, peroxide number and kinematic viscosity were carried out.

5.3 Results and discussion

The properties of the different oils before the experiment are shown in Table 4.

Oil sample	Filtered	Settled
Suspended solids (mg/kg)	13	250
Water content (mass%)	0.055	0.066
Acid value (mg KOH/g)	0.43	0.52
Peroxide value (meq per kg)	2.4	2.4
Kinematic viscosity@ 40°C (mm ² /s)	34.4	34.4

Table 4: Properties of the rape-seed oil at commencement of trial.

5.3.1 Peroxide number

Fig. 11 shows the increase in peroxide number of the rape-seed oil over time in the various samples. The combined effects of high temperature stirring and open-container storage led to a very immediate and rapid increase in peroxide. In contrast, samples stored in airtight conditions at room temperature had shown very little increase even after 70 days. At room temperature, stirring and open-container storage led to only a moderate increase in peroxide level, as did storage at 50°C in airtight conditions. Filtering had little or no effect on the increase in peroxide number.

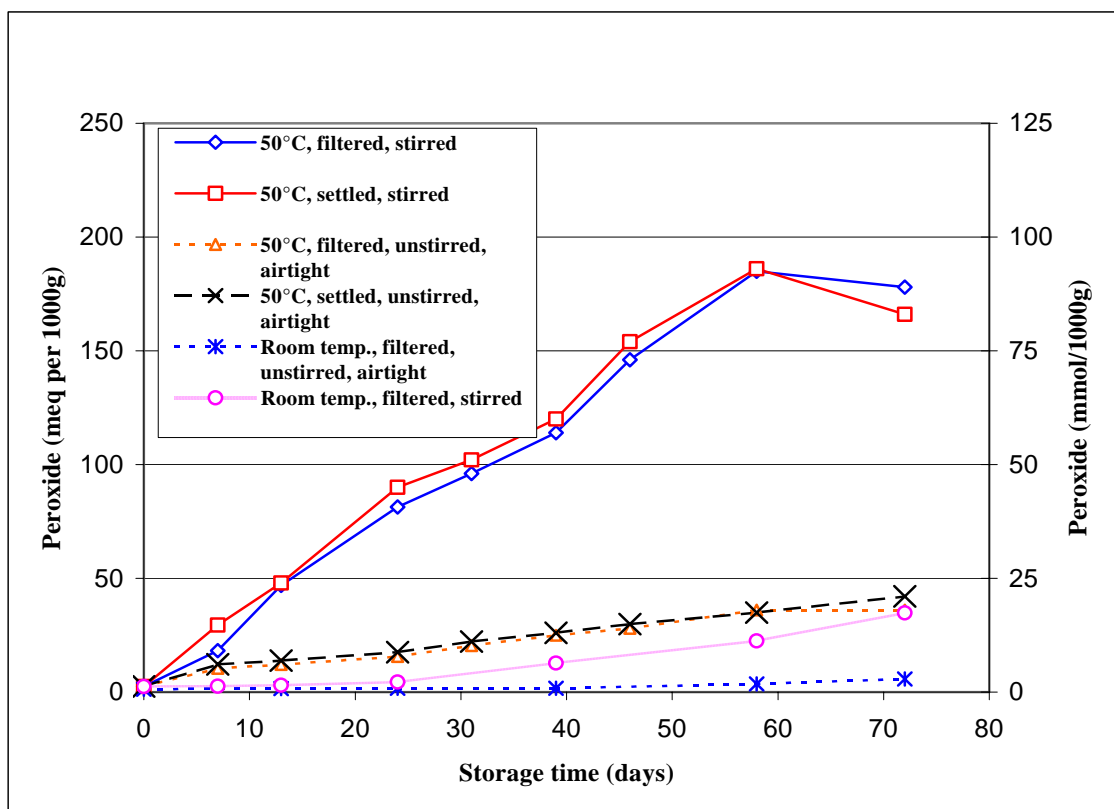


Fig. 11: Increase in peroxide number of rape-seed oil stored in various conditions.

5.3.2 Kinematic viscosity:

Kinematic viscosity reacted slowly to variations in storage conditions (Fig. 12). Little change could be noticed for the first three weeks, by which stage peroxide number in some treatments had increased to 80-100 meq/kg. Thereafter the viscosities of the stirred open-container treatments at 50°C increased steadily and exceeded the RK-limit of 38 mm²/s after about 40 days storage (Fig. 12).

Over the 72-day duration of the experiment there was very little change in the viscosity of any of the other oil samples. All the oils stored at room temperature, and the airtight sample at 50°C, showed only slight increases in viscosity and remained well within the RK limit. Final filtration had very little effect on the increase in viscosity of any of the samples.

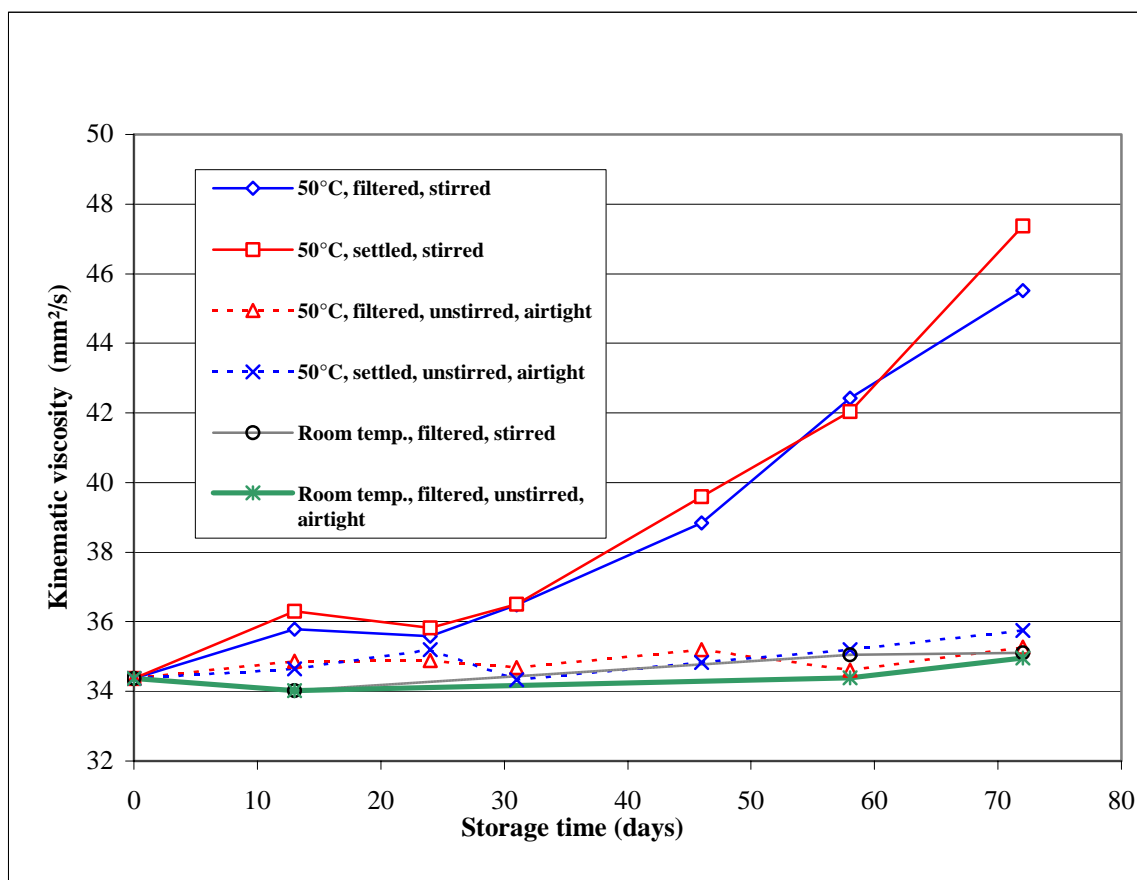


Fig. 12: Increase in viscosity of rape-seed oil stored in various conditions.

5.3.3 Acid value:

All acid values at the end of the trial were still under the RK-limit of 2.0 mg KOH/g. Over the second half of the trial period the level of free fatty acids in the stirred open-container oils at 50°C began to rise sharply. At this stage the peroxide levels in these samples had reached levels between 100 and 150 meq/kg, and their viscosities were also beginning to increase rapidly. It is also noticeable that the level of free fatty acids for the

settled oil was consistently higher than that of the filtered oil. The acid values of the airtight oil at 50°C and all the room-temperature samples showed only very slight increases over the duration of the trial.

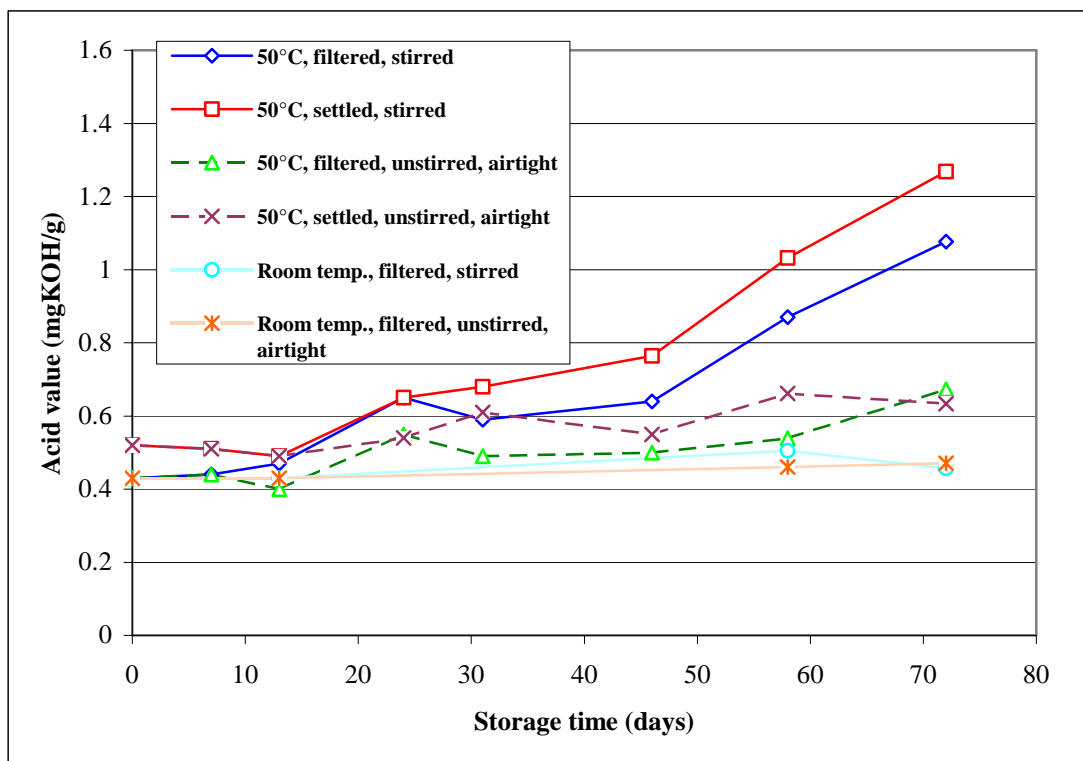


Fig. 13: Increase in acid value of rape-seed oil stored in various conditions.

5.4 Discussion

The trial demonstrated the extent to which increased temperature and exposure to air could accelerate the deterioration of rape-seed oil. Since the temperature of the oil leaving an oil press is likely to be about 50°C, it is important to allow it to cool as quickly as possible. While stirring may be desirable to prevent an accumulation of sediment in the primary storage tank, its role in increasing exposure to air and therefore accelerating deterioration must also be borne in mind.

Filtration of the oil had little effect on peroxide levels or viscosity, but did lead to reduced acid values. Bearing in mind that the oil from press B had a very high level of suspended solids in the first months of storage, this may explain in large measure the high acid values recorded in this oil.

Since most filters operate more efficiently with warm oil, the optimum strategy is to filter the oil as soon as possible after pressing, and then allow it to cool and store in closed containers.

While the viscosity of fresh rape-seed oil is normally well below the RK limit, poor storage conditions may cause it to move above the limit, in extreme cases within one month of pressing. In this case high

temperature and exposure to air are the main problems; solids in the oil appear to have little effect.

While it is not included in the RK standard, these trials show that peroxide level is a good early indicator of oil deterioration in storage. While the acid values and viscosities of the high-temperature, stirred samples showed little increase in the first month of storage, the peroxide levels in these treatments were indicating problems within two weeks of the start of the trial.

6. Conclusions

Several of the oil quality deficiencies reported in this work can be attributed to start-up problems with the plants involved, e.g. excessive delays between pressing and filtration, excessive weed seed contamination, inadequate seed cleaning facilities, and lack of experience in the management of the plants. Nevertheless, the production of oil of consistently acceptable quality for use in modified diesel engines presents a big challenge for small cold pressing and filtration units.

Much remains to be learned about the relative importance of the various oil properties and the limits that should be set for each property to assure a good engine performance when the oil is used as fuel. In the meantime, the RK Standard has been developed on the basis of the best available knowledge at this time, and it should be accepted as the target for cold-pressing plants.

Of the properties listed in the RK Standard, the control of suspended solids and acid value presents by far the biggest problems. To get suspended solids below the RK limit requires the removal of most of the solids by a filter press or several weeks of sedimentation, followed by a pass through a suitable polishing filter. It is also important to ensure that no re-contamination occurs due to solids residues in pumps, pipelines or storage tanks.

For on-site checking of solids levels, the ASG Quick-test kit can be used. The method gives a reasonably clear indication where the RK limit is being exceeded, but it would not guarantee that samples were within the RK limit nor would it give much indication of the extent to which the limit was being exceeded. For the present the best option would be to use the quick-test method on site with occasional verification tests by the laboratories at Oak Park, Carlow Institute of technology or University of Limerick, or from the specialist lab at ASG. It is unlikely that a test method based on the use of a centrifuge would be capable of resolving between samples which are close to the RK limit unless very large sample sizes were used which would make the test very expensive.

While many of the samples in this work had high acid values, small cold-pressing units should be able to achieve the RK limit without much difficulty. The main requirements for the control of acid value are:

- Ensure that the seed is free from all contaminants, especially weed seeds. This requires an effective seed cleaning system, but it also requires a good weed control programme in the production of the crop.
- Keep the period between oil extraction and filtration/cooling to a minimum, preferably less than a week.
- Store with minimum exposure to air.
- Do not allow fresh oil to become contaminated with older oil whose acid value may already have increased.

The ASG Quick test gives quite accurate results for acid value and should be used to make regular on-site checks at every pressing plant. The test should be carried out once weekly on freshly-pressed oil and once monthly on oil in storage.

Of the other process-dependent properties, oxidation stability would be improved by the measures taken to control acid value, and should not be a problem. Phosphorus level should stay within the RK limit as long as the seed is dried to the optimum moisture range and properly cleaned and the press is set to avoid excessive oil temperatures. Ash contents should be kept within limits by the measures taken to control suspended solids (e.g. better filtration, cleaning of oil containers and handling equipment) and acid value (e.g. better

seed cleaning). Water content should not be a problem if the seed is adequately dried and the oil is properly filtered and stored; the ASG quick test provides a simple check that could be carried out at any press site.

All of the characteristic oil properties should remain within the RK limits, and they would only require to be measured if some departure were made from the standard rape-seed oil varieties as an oil source. Viscosity may increase in poor storage conditions, but acid value or peroxide level would be better indicators of this type of problem.

To ensure that high-quality oil is produced consistently, the following weekly records should be made on site:

- Seed moisture and temperature entering the press
- Oil temperature leaving the press
- Total suspended solids, acid value and water content of oil samples removed at exit from the filter press and at the point of despatch. The ASG Quick-test kit could be used for these tests.

Monthly measurements by the appropriate standard methods should be made at an external laboratory of the following:

- Total suspended solids
- Acid value
- Water content (these values for comparison with Quick-test values)
- Phosphorus content
- Ash content
- Oxidation stability

Samples for these analyses should also be removed after the filter press and at the point of despatch. Examples of suitable log sheets are included in Appendix A.

7. References

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Appendix A

Specimen oil quality control sheets for weekly and monthly test records

Oil quality control sheet for weekly checks

Operator									
Limiting Values		<0.075%	<2 mg/KOH/g	<25mg/kg	<60°C	>12°C	6.5-7.5%	Control Section	
Date	Oil Sample Point	Oil Moisture Content	Oil Acid Value	Oil Total Solids	Oil Temp into filters	Seed Temp into Press	Seed Moisture Content	Within Limiting Values Y/N	Comment
	After filters								
	At dispenser								
	After filters								
	At dispenser								
	After filters								
	At dispenser								
	After filters								
	At dispenser								
	After filters								
	At dispenser								
						Signed			
						Date			
<p><i>Instructions: The tests for suspended solids, acid value and water content should be carried out each week using the ASG Quick-test kit and logged as above.</i></p> <p><i>If the parameters including temperature and seed moisture content exceed the limiting values then oil samples should be sent for independent analysis to an external laboratory.</i></p>									

In any event oil samples should be sent at the end of each month for independent analysis. A copy of this record sheet should be included with the samples.

Oil Quality Control Sheet for monthly external checks

Oil Quality Control Sheet for monthly external checks									
Operator									
Limiting Values		<0.075%	<2 mg/KOH/g	<25mg/kg	<15 mg/kg	<0.01%	>5 h	Control Section	
Date	Oil Sample Point	Oil Moisture Content	Oil Acid Value	Oil Total Solids	P content mg/kg	Ash content mass %	Oxidation stability h	Within Limiting Values Y/N	Comment
	After filters								
	At dispenser								
	After filters								
	At dispenser								
	After filters								
	At dispenser								
	After filters								
	At dispenser								
	After filters								
	At dispenser								
						Signed			
						Date			
<p><i>Instructions: The tests for suspended solids, acid value and water content are made to allow comparison between on-site quick-test and external standard tests.</i></p> <p><i>One-litre samples should be taken at the end of each month for external analysis. A copy of this record sheet should be included with the samples.</i></p> <p><i>It is vital that the sample containers be absolutely clean.</i></p>									