

Establishment of the Guidelines for the Development of Biodiesel Standards in the APEC Region

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Hart Energy Consulting



**Asia-Pacific
Economic Cooperation**



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FOREWORD

This project, which was initiated by the Expert Group on New and Renewable Energy Technologies under the APEC 21st Century Renewable Energy Development Initiative Collaborative IX, was an effort of Thailand in close collaboration with Australia, Chinese Taipei, New Zealand, and USA as the project partners to promote the standardization of biodiesel produced in the APEC region.

The project's objective was to establish guidelines for development of biodiesel standards in the APEC region that will enhance biodiesel trade. The use of biodiesel for transportation can reduce air emissions, increase domestic supply of renewable fuel and create new markets for agricultural sectors. Biodiesel fuel can be produced from various animal fats and vegetable oils, but the quality of the product depends on the natural characteristics of the feedstocks used.

The current European biodiesel specification EN 14214 is based on biodiesel produced from rapeseed and combinations of oils that together provide similar characteristics to rapeseed oil, including sunflower, soy and palm. Most of the Asian APEC member economies have derived their biodiesel standards from the European standard, with elements from the U.S. ASTM D6751 biodiesel standard, which was developed to address biodiesel produced predominantly from soy and waste cooking oils. Although Europe is not an APEC economy, it has increasing requirements to reduce CO₂ emissions, coupled with a growing shortage of diesel fuel. Thus, Europe represents a large potential biodiesel export market for APEC producer economies.

Economies in the Asia Pacific have different climates, feedstocks and vehicle fleets, which may require a different biodiesel standard in order to ensure market acceptance and to maximize the opportunity for regional trade. This study compares current biodiesel standards between APEC member economies, Europe, Brazil and India and outlines the key biodiesel quality parameters required to ensure the durability and performance of vehicles in the APEC region.

With this note, I would like to thank Hart Energy Consulting for a job well done of the study. I must also pay tribute to those individuals and organizations in the APEC economies who provided additional information and contributed to the successful completion of the report.

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1. Executive Summary

1.1. Background and Objective

Hart Energy Consulting was contracted to draft a position paper establishing guidelines for the development of a performance-based common biodiesel standard to enhance the trade of biodiesel among the twenty-one Asia Pacific Economic Cooperation (APEC) member economies.

For this study, the term “biodiesel” refers to so-called 1st generation biodiesel and is defined as a fatty acid alkyl ester, most typically produced by transesterification of triglycerides with methanol (forming fatty acid methyl ester, or FAME), and with glycerol as a co-product. Tri-glyceride feedstocks include vegetable oils and animal fat tallows. Straight vegetable oils, that are generally of too poor stability and have poor cold flow properties, and so-called 2nd generation bio-based diesel substitutes are excluded. 2nd generation biodiesels are distillate fractions that boil in the diesel range, and have qualities that are in some cases superior to conventional crude oil derived diesel.

Asia boasts a wide variety of animal fats and vegetable oils suitable for biodiesel production, including waste cooking oil, palm, coconut, jatropha, rapeseed and soy oils, and tallow from many animals. In fact, Malaysia and Indonesia are the world’s largest palm oil producers, The Philippines is the world’s largest coconut producer and exporter and China is the world’s leading producer of rapeseed. The United States (U.S.) is a significant producer of corn, soy and other crops. Thailand and Viet Nam, being agricultural-based economies, are able to grow a variety of suitable crops. However, APEC economies (such as: Brunei Darussalam; Hong Kong, China; Japan; Singapore; Korea¹; Chinese Taipei and others) are likely to depend on biofuel imports due to insufficient suitable land to produce both food and biofuels. Hence, the opportunity for a regional standard to facilitate trade in biodiesel amongst the APEC member economies is apparent.

This study compares the current biodiesel standards between APEC member economies, Europe and Brazil, and outlines the key biodiesel quality parameters required to ensure the durability and performance of vehicles in the APEC region.

1.2. Biodiesel Specification Developments and Experience

To date, very little formal co-ordination or information sharing has occurred between the world’s economies with respect to their biodiesel standards. Some standards are based on B100 use, some on B100 used only for blending B5 or B20. Additionally, some standards are more prescriptive specifying compounds, while others are focussed on the specification of performance properties making them more generic and feedstock neutral.

¹ Based on the APEC accepted nomenclature. In this report 'Korea' refers to 'South Korea.'

The current European biodiesel specification, EN 14214, is primarily based on FAME produced from rapeseed. Use varies from B5, without labelling, to B30 to B40 in captive fleets and B100 from retail sites to vehicles with B100 approvals. The B5 must meet the EN 590 automotive diesel fuel standard. A specific blend specification for higher levels than B5 is under deliberation at present.

The U.S. ASTM D6751 biodiesel standard was developed to address FAME biodiesel generically, but the data used to develop the standard has come primarily from biodiesel produced from soy and waste cooking oils. If B100 meets ASTM D6751 and petroleum diesel fuel meets ASTM D975, there is no separate set of specifications for the blend. In the U.S., most of the experience with biodiesel has been with B20, with market share of diesel/biodiesel blends being approximately 75% B20, 24% B5/B2 and 1% B100. The use of B20 has been the subject of annual quality and stability surveys and few operational problems are experienced. Most of the established problems are usually caused by biodiesel not meeting the requirements of ASTM D6751 (high glyceride or soap content) or other issues (cleaning effect of biodiesel, poor cold flow, microbial growth, improper blending).

The Brazilian biodiesel standard, Agência Nacional de Petróleo (ANP) Act No.42, finalized in 2004, allows for much greater biodiesel feedstock diversity than the EN and U.S. standards. This standard classifies properties related to the “quality of the process” versus those related to the “nature of the raw materials” (the feedstock). No limits are set for properties where either the analytical method (for multi-feedstock) or the actual limit is still undefined due to the diversity of raw materials, and where no performance issues are anticipated. The Brazilian philosophy to biodiesel specifications could be described as recognizing biodiesel as a blend component, for which the properties may not comply with the end market specification, but can be used via blend synergy to formulate a diesel that does comply with performance requirements. This is the same approach taken in a conventional oil refinery, where marketable diesel is blended from a range of characterized diesel blend components that individually may not comply with the final product specification.

1.3. Biodiesel Properties

While biodiesel is generally considered to be a substitute for all or part of diesel fuel, there are some key differences that need to be considered when examining pure biodiesel replacing diesel or when it is blended with diesel. Key differences are:

- Biodiesel is a combination of a small range of molecules, typically esters of one of a number of fatty acids of C₁₂, C₁₄, C₁₆, C₁₈ and C₂₂, whereas diesel is a complex mixture of a broad range of hydrocarbons from C₁₂ to C₂₅, consisting of paraffins, naphthenes and aromatics, as well as a range of nitrogen and sulfur containing organic compounds;
- Diesel is produced largely by distillation of a broad cut of petroleum, to separate the lighter (lower flash point gasoline and kerosene) and heavier (typically waxy, fuel oils and heavier distillates) components, whereas biodiesel is produced by a chemical reaction followed by physical separation.

Biodiesel thus may contain some heavy materials or materials that are subject to thermal decomposition when exposed to heat occurring in an engine;

- Biodiesel may contain a high level of unsaturated (olefinic) carbon bonds. Normal crude oil contains few olefins and refinery hydrotreatment, meant to reduce sulfur, further lowers this. Olefins are unstable and may contribute to deposits and higher rates of degradation; and
- Biodiesel contains oxygen, as the ester, which fundamentally affects its performance properties. It reduces energy content and makes the biodiesel polar, through the hydroxyl (-OH) hydrogen bond. The polarity gives properties of solvency, detergency, wet-ability (sticking to metals as a lubricant) and conductivity.

Pure biodiesel has excellent anti-foam properties, better than petroleum diesel.

Cetane number (CN) is one of the most common indicators of diesel fuel quality. The CN of biodiesel is generally between 45 and 70, as compared to 40 to 52 for typical diesel fuels.

For diesels, each component has its own crystallization temperature, so solidification is a gradual process, whereas B100 biodiesel tends to be a much simpler mixture containing relatively few components, so that one or two components tend to dominate, and solidification is much more rapid and difficult to control. The temperature at which B100 starts to gel varies significantly depending on the mix of esters and therefore on the feedstock oil used to produce the biodiesel.

Some biodiesels are more susceptible to oxidation than diesels and tend to produce gums and lacquers, creating potential for significant increases in deposits. Modern diesel direct injection engines meeting the European Euro IV emission standards have smaller injectors operating at higher pressures and hence are more susceptible to deposits. Specific oxidation inhibitors are therefore required, and the European industry has moved to antioxidant treatment as part of the biodiesel manufacturing process. Biodiesel and biodiesel blends respond well to diesel dispersant additives to control deposits. With regard to specific requirements for maximum residue, the CCR test, as developed for diesel and other petroleum products, has been utilized successfully for biodiesel. In fact, it is important for biodiesel as it indicates impurities, such as free fatty acids, glycerides, soaps, insolubles, polymers, additives like pour point depressants and content of higher unsaturated fatty acids.

Ultimately, the quality of biodiesel is a function of:

- feedstock qualities,
- process, including the control of the process (the expression “built in quality” applies)

The transesterification process works on all kinds of triglycerides, but some characteristics of the ester are directly linked to the properties of the feedstock oil, namely:

- Density; viscosity; cold filter plugging point (CFPP); 90% distillation

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- These are determined by the molecular weight (number of C-atoms) – being lower for less carbon atoms; branching – lower for more branched molecules; and degree of saturation, with unsaturation leading to better cold temperature properties.
- Cetane number
 - Longer and straighter chains generally impart high cetane, but this falls as the degree of unsaturation rises.
- Iodine number (IN)
 - This is a measure of unsaturation (olefins), as iodine reacts with the unsaturated bonds. A higher number would imply poorer stability, but not necessarily as the higher IN product could have good quality anti-oxidants (AO) added to address instability.
- Oxidation stability
 - This is better for more saturated (less olefinic) triglycerides. As the number of double bonds rises, the propensity to oxidize in the presence of air rises, leading to instability, so that more AO needs to be added.

The critical quality aspects of the biodiesel production process are:

- Complete reaction to consume the triglycerides
 - Needed to comply with specification on minimum ester content, mono-, di- and tri-glyceride content, and carbon residue;
- Removal or separation of glycerol (glycerin) co-product
 - Needed to comply with specification on free and total glycerol and carbon residue;
- Removal of catalyst (Na and K)
 - These are limited in biodiesel specifications due to possible interaction with emission control devices and may also cause deviation of the sulfated ash limit;
- Removal of alcohol (typically methanol)
 - Needed to comply with limits on flash point and methanol content;
- Separation of free fatty acids (FFA)
 - Needed to comply with acid number. High acid numbers lead to instability and incompatibility with fuel system metals, and will not pass the copper corrosion test;
- Removal of water (drying)
 - Needed to avoid fuel injection system corrosion;
- Cleanliness and filtration
 - Total contamination compliance to avoid filling station and vehicle filter blocking, and premature wear of injection systems.

1.4. Additives

The main additive companies supplying petroleum diesel additives and vegetable oil additives include Lanxess, Ciba, Eastman, Lubrizol, Degussa, Afton Chemical Corporation, BASF and Innospec. These companies have over recent years developed

and proven additives for biodiesel and biodiesel blends, and have generated test results that they willingly share. However, most market experience has been with rapeseed methyl ester, as this is the highest volume of biodiesel globally, and with soya methyl ester.

Biodiesel producers and marketers need to ensure that their biodiesel and the diesel blends thereof are tested with the recommended additives to confirm the resultant property improvements. In addition, particularly where higher treat rates are used than applies to conventional diesel, “no harm” testing is required. This should be backed by market monitoring for initial introductory periods.

Further work is required to ensure that biodiesel and biodiesel blends can comply with market demands. The availability of suitable additives for wider ranges of feedstocks, and test methods for petroleum based fuels that correlate with field performance should also apply to biodiesel.

Two of the major obstacles to widespread acceptance of higher biodiesel blends are:

- fuel stability, and
- low temperature operability.

These can be addressed by a combination of feedstock selection, proper processing and quality control, as well as by the use of appropriate fuel additives, particularly for stability. It is important to note that fuel additives have the most benefit at an optimal dosage rate for each application, beyond which saturation is reached. Dosage exceeding saturation levels can lead to decreased performance results.

1.5. Vehicles Compatibility and Emissions

Historically it was intended to produce biodiesel in a closed-loop from agricultural products to agricultural machinery, mainly tractors, and the farming sector promoted its use with equipment suppliers. Hence, the first warranties issued were for tractors or Combines (e.g. Same, Steyr, John Deere, Massey-Ferguson, Lindner etc.). With the development of more sophisticated marketing strategies, the focus has extended to include other diesel-driven vehicles such as buses in the public transport fleets of cities, taxi fleets, the coastguard, and more sensitive users including cruise ships, the marine leisure sector, underground mines, and certain private car owners. Private cars using biodiesel increased significantly after warranties for the new generation of modern, high-pressure fuel injection systems such as the common-rail systems (e.g. Mercedes-Benz, Peugeot, and Volkswagen (VW)) were obtained. Today more than 400,000 cars use pure biodiesel (B100) in Germany, Austria and Sweden. In contrast, 10 to 15 million cars use biodiesel as an extender (B2) to fossil diesel fuel in France. A “middle of the road” approach is the strategy adopted by Chechnya and Slovakia, and also in France, which offers fossil diesel blended with 30 to 40 vol% biodiesel in addition to B2 blends.

Engine manufacturers have two primary areas of concern:

1. stability and quality of existing biodiesel; and

2. effect of biodiesel combustion on the emissions and performance of their existing engines, and on the emerging advanced combustion regime engines they are designing to meet more stringent emissions requirements.

The Fuel Injection Equipment (FIE) manufacturers (not the vehicle manufacturers) warranty their hardware in the vehicle. To date, the FIE manufacturers only accept the EN 14214 biodiesel specification.

The World Wide Fuel Charter, representing the automobile and engine manufacturers desired fuel specifications, indicates that automotive manufacturers prefer not to have biodiesel in the latest technology vehicles designed for the lowest emissions and requiring category 4 fuels. This is due to uncertainties with regard to the compatibility between biodiesel and the most advanced emission control devices. The EU legislators however require the use of blends up to B5 in such vehicles. At higher blend levels, engine, auto and fuel injector manufacturers have concerns linked to instability and the presence of oxidation products in biodiesel. Some fuel injection equipment test data suggests that such stability problems may be exacerbated when biodiesel is blended with ultra-low sulfur diesel fuels (<10 ppm sulfur), as it is known that the natural antioxidants are often removed by the refinery desulphurization process, leading to lower stability in the diesel fuel itself. This can generally be overcome by the addition of appropriate antioxidants, as is the recommended practice now in Europe. Generally, a B5 blend to a suitable specification is not expected to cause problems and can be relatively aggressively adopted.

Blends of biodiesel from B5 to B20 could have some engine and fuel system compatibility issues, and injector nozzle coking tendencies. Currently, very few manufacturers have certified vehicles for use with these blend levels in a limited number of their product lines. It is expected that the availability of such blends would require additional labeling requirements and consumer awareness of possible compatibility issues. A separate specification for the B20 blend itself will probably also be required. However, most new vehicles sold today are expected to be compatible, with problems, if any, only arising in pre-model year 1996 vehicles.

Blends above B20 are currently not recommended by most manufacturers. There is limited data available on compatibility and performance related issues with these blends over the useful vehicle life. These blends are expected to require biodiesel compatible equipment and modifications to engine timing to maintain performance.

Emissions reports regarding biodiesel are inconsistent. There is a link between emissions and duty-cycle, type of engine, and condition of the engine. However, there are as yet no known issues with any after-treatment systems being adversely affected by the use of biodiesel that meets either the ASTM D6751 or EN 14214 specification. The U.S. EPA has surveyed a large body of biodiesel emissions studies and the results are shown in Table 9 of this report.

Most biodiesel emission studies have been carried out on existing heavy-duty highway engines. The effects of biodiesel on emissions from heavy diesel engines meeting EPA's stringent Tier II emissions standards (introduced since model year 2007) have not been determined, and the EPA has concluded that the results of biodiesel tests in

heavy-duty vehicles cannot be generalized to light-duty diesel vehicles or off-highway diesel engines. The greenhouse gas (CHG) and specifically carbon dioxide (CO₂) emission benefits are clear and desirable, and the inherently favorable chemical composition, which includes oxygen in the fuel molecules, will directionally reduce hydrocarbon (HC) and particulate matter (PM) emissions.

The transition period in automobiles from diesel to biodiesel blends that have higher solvency may lead to an especially large increase in sediments that could plug fuel filters. It is recommended by most vehicle manufacturers that filters are changed as a precautionary measure after the first tank of fuel containing biodiesel has been used.

1.6. APEC Biodiesel Standard

Experience has shown that biodiesel market problems often have less to do with the standards, and more to do with poor manufacturing practices and quality control. For this reason, effort should be focused on ensuring a standard that is workable for biodiesel producers, and this needs to be backed by a quality control and assurance program that supports high levels of compliance. A proper reporting and monitoring program is therefore key to successful national biodiesel programs.

The APEC economies have tended to follow a process where a biodiesel standard is set using either the ASTM D6751 or EN 14214 as a guideline with adaptation to local conditions. Canada allows up to 1% biodiesel as an additive under the existing diesel fuel specification. The standard for B1 to B5 fuel (CAN/CGSB 3.520, 2005) uses the existing low sulfur and ultra-low sulfur diesel specifications, and requires the biodiesel that is blended to meet either the ASTM D6751 or the EN 14214 specification.

In developing an APEC biodiesel standard, a similar approach may be followed, including:

- agreeing on a minimum “fit for purpose” standard as soon as possible, perhaps even without all the answers;
- defining and conducting research programs to examine the chemically based parameters of the standard that restrict certain feedstocks and try to replace them with less restrictive performance based parameters accompanied by suitably defined test methods;
- providing quality assurance programs for both manufacturers and fuel blenders;
- providing field experience monitoring; and
- establishing a framework for adjustments and advancements as necessary.

At the first workshop to discuss the establishment of the guidelines for development of a biodiesel standard in the APEC region, held in Thailand on October 25 and 26th, 2007, it was agreed by those APEC member economies and experts present that:

- the standard should be based on 1st generation biodiesel;
- it should be developed for B100 as a trading fuel, which is to be blended according to local vehicle conditions and fuel standards of APEC member economies. At present the U.S. has vehicles that require sophisticated

- emissions control systems to meet stringent tier 2 emissions regulations, whereas many of the APEC economies in Asia (excluding Japan, Australia and others) have vehicles that are not equipped with particulate traps and oxygen sensors as they do not as yet have emissions regulations that require them;
- it should focus on the best way to facilitate the largest volume of trade, which translates to lower percentages of biodiesel sold to a greater number of vehicles. Therefore the standard should be developed for high speed vehicles;
 - largely due to initial availability of biodiesel and suitable logistics systems, the end use would likely be B5 during the early years, with the blended fuel meeting the existing local diesel standard;
 - the same biodiesel can be blended at higher levels, such as B10, B20 and B50. These higher blends may require waivers from the diesel specification, or may require special standards. In such cases, due to being tailored to specific user needs, for captive fleets, or where the higher blend, including B100, is sold to the public, this should include specific communication such as product labelling;
 - if biodiesel that did not meet the future APEC B100 standard was sold, then it would have to be a non-compliant blend component, with contractual waivers to the APEC specification that had been agreed between buyer and seller.

A review of specific parameters in the APEC economies that have set biodiesel standards showed that there was already agreement on a number of critical parameters. No parameters were identified where it was considered impossible to reach an agreement. Further discussion and research is required to address those areas of difference, primarily:

- flash point, density, viscosity
- max T90 distillation or minimum ester content ?
- phosphorus and other metals; copper corrosion
- cold filter plugging point
- conradson carbon residue; total contamination
- water content specification and test methodology
- total glycerin; iodine number; acidity
- chemical compounds

These parameters are discussed in detail in Chapter 5 of the report.

Expert stakeholders from the APEC economies will also need to:

- conduct an assessment of testing facilities and laboratories in member economies. The representative from Indonesia mentioned for example that there is no laboratory in Indonesia capable of testing all the biodiesel

- parameters in the ASTM D6751 or EN 14214 standards;
- establish accredited test facilities for round-robin testing between APEC economies;
- review all available test data for feedstock dependant variables, and identify further research work required in support of performance based specifications;
- Include the FIE manufacturers in further discussion

APEC expert stakeholders are encouraged to examine and debate the data presented in this report with a view to reaching consensus on the quality control parameters required for an acceptable performance based B100 biodiesel standard to be adopted by APEC member economies.

Table of Contents

1. EXECUTIVE SUMMARY	III
1.1. BACKGROUND AND OBJECTIVE	III
1.2. BIODIESEL SPECIFICATION DEVELOPMENTS AND EXPERIENCE	III
1.3. BIODIESEL PROPERTIES	IV
1.4. ADDITIVES	VI
1.5. VEHICLES COMPATIBILITY AND EMISSIONS	VII
1.6. APEC BIODIESEL STANDARD.....	IX
2. ABBREVIATIONS AND ACRONYMS.....	XVII
3. INTRODUCTION.....	1
4. BACKGROUND.....	4
4.1. HISTORY OF BIODIESEL	4
4.1.1. <i>Early Developments with Straight Vegetable Oil</i>	4
4.1.2. <i>Esterification from the 1990s</i>	4
4.1.3. <i>European Leadership</i>	6
4.1.4. <i>U.S. Developments</i>	6
4.1.5. <i>Considerations by OEMs and APEC Needs</i>	6
4.2. DEVELOPMENT OF EUROPEAN, U.S. AND BRAZILIAN BIODIESEL SPECIFICATIONS.....	7
4.2.1. <i>Europe</i>	9
4.2.2. <i>U.S.</i>	10
4.2.3. <i>Brazil</i>	11
4.3. SECOND GENERATION BIODIESEL	12
4.4. BIODIESEL COMPARED TO PETROLEUM DIESEL	14
4.5. SOURCES OF FEEDSTOCK IN APEC ECONOMIES	14
4.6. DIESEL VEHICLES IN APEC ECONOMIES.....	16
4.7. SPECIFICATION, QUALITY CONTROL AND MONITORING APPROACHES.....	16
4.7.1. <i>Market Experience and Quality Levels</i>	16
4.7.2. <i>Consumer Needs and Market Impacts</i>	17
4.7.3. <i>Quality Program Needed</i>	17
5. BIODIESEL PROPERTIES.....	18
5.1. PRODUCTION EFFECTS	18
5.1.1. <i>Feedstock</i>	18
5.1.1.1. <i>Free Fatty Acid</i>	20
5.1.1.2. <i>Insolubles</i>	20
5.1.1.3. <i>Iodine Value</i>	21
5.1.1.4. <i>Phosphorus</i>	22
5.1.1.5. <i>Stability and Deposits</i>	23
5.1.1.6. <i>Sulfur</i>	25
5.1.1.7. <i>Water</i>	25
5.1.1.8. <i>Other Contaminants</i>	26
5.1.2. <i>Addressing Feedstock Effects (Shortcomings)</i>	27
5.2. PROCESS DEPENDENT PROPERTIES.....	27
5.2.1. <i>Reaction Completion</i>	29
5.2.2. <i>Washing</i>	29
5.2.3. <i>Other Ester Treatments</i>	30
5.2.4. <i>Glycerol Fraction Separation, Properties and Processing</i>	31
5.3. BIODIESEL AS A PERFORMANCE FUEL	31
5.3.1. <i>Antifoaming</i>	32
5.3.2. <i>Cetane Number</i>	32
5.3.3. <i>Chemical Structure and Oxygen Content</i>	32

5.3.4.	<i>Cold Flow Properties</i>	33
5.3.5.	<i>Conductivity</i>	33
5.3.6.	<i>Corrosion</i>	34
5.3.7.	<i>Density</i>	34
5.3.8.	<i>Viscosity</i>	35
5.4.	BLEND WITH DIESEL & ADDITIZATION.....	36
5.4.1.	<i>Antifoaming</i>	37
5.4.2.	<i>Cetane</i>	37
5.4.3.	<i>Cold Flow Properties</i>	37
5.4.4.	<i>Conductivity</i>	37
5.4.5.	<i>Density</i>	38
5.4.6.	<i>Lubricity</i>	38
5.4.7.	<i>Viscosity</i>	38
5.4.8.	<i>Stability (Oxidation)</i>	38
5.4.9.	<i>Performance Additives</i>	39
5.4.9.1.	Corrosion Protection (Inhibitors).....	39
5.4.9.2.	Injector Deposits (Detergents).....	40
5.4.9.3.	Water Separation (Demulsifiers).....	40
6.	VEHICLES COMPATIBILITY AND EMISSIONS	41
6.1.	DIESEL VEHICLE REQUIREMENTS.....	42
6.1.1.	<i>Fuel System Effects</i>	42
6.1.1.1.	Elastomers.....	42
6.1.1.2.	Filters.....	43
6.1.1.3.	Injector Systems.....	43
6.1.1.4.	Metals.....	44
6.1.1.5.	Paint.....	44
6.1.2.	<i>Engine Performance</i>	44
6.1.2.1.	Cold Flow.....	46
6.1.2.2.	Cylinder heads.....	46
6.1.2.3.	Engine Oil.....	46
6.1.2.4.	Fuel Economy.....	46
6.1.2.5.	Lubricity.....	47
6.1.2.6.	Maintenance Cost.....	47
6.1.2.7.	Piston.....	47
6.1.2.8.	Power/Torque.....	47
6.1.2.9.	Injection Timing.....	48
6.1.3.	<i>Emissions</i>	48
6.1.3.1.	Carbon Monoxide.....	50
6.1.3.2.	Hydrocarbons.....	50
6.1.3.3.	Nitrogen Oxides.....	50
6.1.3.4.	Ozone.....	51
6.1.3.5.	Particulate Matter.....	51
6.1.3.6.	Polycyclic Aromatic Hydrocarbons.....	52
6.1.3.7.	Sulfur.....	53
6.1.3.8.	Catalyst Deactivation.....	53
6.1.3.9.	Diesel Particulate Filter Systems.....	53
6.2.	GAPS AND UNCERTAINTIES.....	53
6.3.	POSSIBLE PROBLEMS AND ACCEPTANCE.....	53
6.4.	RELEVANCE FOR APEC VEHICLES.....	55
7.	APEC BIODIESEL STANDARD.....	57
7.1.	APPROACH.....	57
7.2.	SPECIFICATION PARAMETERS.....	58
8.	CONCLUSIONS.....	67

9. REFERENCES.....	69
10. APPENDICES.....	73
10.1. GLOBAL BIODIESEL SPECIFICATIONS	74
10.2. FEEDSTOCK AVAILABILITY AND BIOFUELS POTENTIAL IN THE APEC REGION	79
10.2.1. Australia.....	79
10.2.2. China.....	80
10.2.3. Chinese Taipei	81
10.2.4. India	81
10.2.5. Indonesia.....	81
10.2.6. Japan.....	82
10.2.7. Korea	82
10.2.8. Malaysia.....	82
10.2.9. New Zealand	83
10.2.10. The Philippines	83
10.2.11. Thailand.....	83
10.2.12. U.S.....	83
10.3. APEC REGIONAL EXPERIENCE WITH PRODUCTION AND STANDARDS	84
10.3.1. Australia.....	84
10.3.2. Japan.....	85
10.3.3. Malaysia.....	86
10.3.4. Philippines	87
10.4. VEHICLE FLEET INFORMATION BY ECONOMY	87
10.4.1. Australia.....	87
10.4.2. Japan.....	89
10.4.3. Malaysia.....	93
10.4.4. New Zealand	95
10.4.5. The Philippines	98
10.4.6. U.S.....	100
10.5. QUALITY CONTROL ASPECTS ⁰	104
10.5.1. BQ 9000 Quality Program.....	106
10.5.2. BQ-9000 Accredited Marketer	107
10.5.3. Key Issues for Controlling Biodiesel Consistency and Quality.....	108
10.6. STORAGE AND HANDLING EFFECTS.....	109
10.6.1. Storage Stability.....	109
10.6.2. Comparatively Low Toxicity to Marine Plants and Animals	110
10.6.3. Biodegradability of Biodiesel in the Aquatic Environment.....	110
10.7. STANDARDS AND SPECIFICATIONS	110
10.8. DIESEL FIE MANUFACTURERS COMMON POSITION STATEMENT ON FAME FUELS	111

List of Figures

FIGURE 1: GEOGRAPHIC INTRODUCTION OF SECOND GENERATION BIODIESEL.....	2
FIGURE 2: GLOBAL VIEW OF BIODIESEL USAGE, MARCH 2007.....	8
FIGURE 3: GLOBAL BIODIESEL BLENDING LIMITS, APRIL 2007	8
FIGURE 4: COMPOSITION OF VARIOUS BIODIESEL FEEDSTOCK	15
FIGURE 5: SCHEMATIC OF THE TRANSESTERIFICATION PROCESS TO PRODUCE BIODIESEL.....	28
FIGURE 6: AVERAGE EMISSION IMPACTS OF BIODIESEL FOR HEAVY-DUTY HIGHWAY ENGINES	49
FIGURE 7: BIODIESEL SOURCE EFFECT ON CO FOR HEAVY-DUTY DIESEL ENGINES	50
FIGURE 8: BIODIESEL SOURCE EFFECT ON NO _x FOR HEAVY-DUTY DIESEL ENGINES.....	51
FIGURE 9: BIODIESEL SOURCE EFFECT ON PM FOR HEAVY-DUTY DIESEL ENGINES	52
FIGURE 10: 2006 DIESEL FLEET COMPOSITION BY TYPE IN AUSTRALIA.....	88
FIGURE 11: RATIO OF VEHICLE FLEET BY FUEL TYPE IN AUSTRALIA.....	89
FIGURE 12: VEHICLE POPULATION AND SCRAPPAGE RATE IN JAPAN.....	90
FIGURE 13: IN-USE DIESEL VEHICLES, 2006 FLEET DISTRIBUTION IN JAPAN	91
FIGURE 14: IN-USE DIESEL VEHICLES BY TYPE IN JAPAN, 2006.....	92
FIGURE 15: FLEET DISTRIBUTION OF NEW VEHICLES IN 2006.....	93
FIGURE 16: VEHICLE POPULATION AND SCRAPPAGE RATE IN MALAYSIA	94
FIGURE 17: FLEET DISTRIBUTION OF NEWLY REGISTERED VEHICLES IN MALAYSIA, 2006.....	95
FIGURE 18: DISTRIBUTION OF DIESEL VEHICLES BY AGE IN NEW ZEALAND, 2006	96
FIGURE 19: DISTRIBUTION OF TRUCKS AND BUSES BY AGE FOR NEW ZEALAND, 2006	97
FIGURE 20: DIESEL VEHICLE DISTRIBUTION BY TYPE AND SCRAPPAGE RATE FOR NEW ZEALAND, 2006.....	98
FIGURE 21: VEHICLE POPULATION AND SCRAPPAGE RATE FOR THE PHILIPPINES	99
FIGURE 22: FLEET DISTRIBUTION OF NEWLY REGISTERED VEHICLES IN THE PHILIPPINES, 2006 ...	100
FIGURE 23: LIGHT DUTY VEHICLE POPULATION AND SCRAPPAGE RATE FOR U.S.....	101
FIGURE 24: MEDIAN AGE OF VEHICLES IN THE U.S.	102
FIGURE 25: FLEET DISTRIBUTION OF REGISTERED VEHICLES IN U.S., OCT, 2006	103
FIGURE 26: NEW VEHICLE REGISTRATION TREND IN THE U.S.....	104

List of Tables

TABLE 1: BRAZILIAN APPROACH TO BIODIESEL SPECIFICATION PARAMETERS	12
TABLE 2: FUEL PROPERTIES AS A FUNCTION OF FUEL COMPOSITION IN DIESEL ENGINES.....	15
TABLE 3: SATURATION LEVELS OF DIFFERING FEEDSTOCKS.....	19
TABLE 4: OILS AND THEIR MELTING POINT AND IODINE VALUE.....	21
TABLE 5: TYPICAL PROPERTIES OF BIODIESEL COMPARED TO DIESEL	34
TABLE 6: BIODIESEL BLENDS COMPATIBLE WITH FIE MANUFACTURERS AND THEIR QUALITY REQUIREMENTS	42
TABLE 7: BIODIESEL BLENDS APPROVED FOR USE BY MANUFACTURERS AND THEIR QUALITY REQUIREMENTS	44
TABLE 8: BRAND TYPE MODELS WITH WARRANTIES FOR B100	45
TABLE 9: AVERAGE BIODIESEL EMISSIONS COMPARED TO DIESEL.....	49
TABLE 10: PROPERTIES OF EUROPEAN RAPESEED BIODIESEL THAT CAN CAUSE VEHICLE PROBLEMS	54
TABLE 11: BRAZILIAN APPROACH TO BIODIESEL SPECIFICATIONS, ADDING EUROPEAN AND ASTM AND APEC NATIONAL SPECIFICATION REQUIREMENTS TO FACILITATE DISCUSSION OF PARAMETERS FOR APEC STANDARD.....	59
TABLE 12: APEC BIODIESEL QUALITY STANDARD AND HARMONIZATION INITIATIVE.....	63
TABLE 13: BIODIESEL POSSIBLE PROBLEMS ADDRESSED BY SPECIFICATIONS IN JAPAN	85

2. Abbreviations and Acronyms

AAM	Alliance of Automobile Manufacturers
ACEA	Association of European Vehicle Manufacturers
AGQM	Arbeitsgemeinschaft umfasst Biodieselhersteller und Vermarkter - Biodiesel Quality Management Association, Germany
AME	Acid Methyl Ester
ANP	Agência Nacional de Petróleo (Brazil's National Petroleum Agency)
APEC	Asia-Pacific Economic Cooperation
ASTM	American Society for Testing and Materials
B100	100% biodiesel
BAC	Biodiesel Association of Canada
BTL	Biomass to liquids
Bx	"x" vol% biodiesel, 100-x vol% petroleum diesel
Ca	Calcium
CCR	Conradson Carbon Residue
CEN	Comité Européen de Normalisation (European Committee for Standardization)
CFPP	Cold filter plugging point
CI	Compression ignition
CME	Coconut methyl ester
CN	Cetane number
CO	Carbon monoxide
CO ₂	Carbon dioxide
CPO	Crude Palm Oil
CRFA	Canadian Renewable Fuels Association
CTL	Coal to liquids
DIN	Deutsches Institut für Normung (German Institute for Standards)
DOE	U.S. Department of Energy
DPF	Diesel particulate filter
EFTA	European Free Trade Association between Iceland, Liechtenstein, Norway and the EU Member States
EMA	Engine Manufacturers Association
EN	European Norm or standard
EPA	U.S. Environmental Protection Agency
EU	European Union
Europe	EU and EFTA economies
FAEE	Fatty acid ethyl ester
FAMAE	Fatty acid mono alkyl ester
FAME	Fatty acid methyl esters
FCAI	Federal Chamber of Automotive Industries
FFA	Free Fatty Acid
FIE	Fuel injection equipment
FT	Fischer-Tropsch
GHG	Green house gases

GTL	Gas to liquids
HC	Hydrocarbon
HFRR	High frequency reciprocating rig, used to test diesel lubricity
IRS	Internal Revenue Service
IV	Iodine value
JAMA	Japanese Automobile Manufacturers Association
JME	Jatropha Methyl Ester
K	Potassium
KOH	Potassium hydroxide
LPG	Liquefied Petroleum Gas
Mg	Magnesium
MPOB	Malaysian Palm Oil Board
MSDS	Material safety data sheet
Na	Sodium
NaOH	Sodium hydroxide
NBB	National Biodiesel Board
NO _x	Nitrogen oxides
nPAH	Nitrated Polycyclic aromatic hydrocarbons
NREL	National Renewable Energy Laboratory
OBD	On-board diagnostics
OEM	Original equipment manufacturer
PAH	Polyaromatic hydrocarbons
PM	Particulate matter
PME	Palm methyl ester
ppm	Parts per million
PV	Peroxide value
RME	Rapeseed methyl ester
SME	Soy methyl ester
SO ₂	Sulfur dioxide
TME	Tallow Methyl Ester
U.S.	United States of America
ULSD	Ultra low sulfur diesel
VOC	Volatile organic compound
VOME	Vegetable oil methyl ester
WWFC	World Wide Fuel Charter of global automakers

3. Introduction

Hart Energy Consulting has been contracted to draft a position paper establishing guidelines for the development of a performance – based common biodiesel standard⁽²⁾ to enhance the trade of biodiesel among the twenty-one Asia Pacific Economic Cooperation (APEC) member economies.⁽³⁾

Maintaining biodiesel product quality is essential for the growth of the biodiesel industry. To this end, the definition of what biodiesel is and is not is very important in discussions regarding its quality and acceptability.

Pure vegetable oils are not defined as “biodiesel” and are usually considered unsuitable for use in engines due to their poor cold flow properties, high viscosity, low cetane, low stability and tendency towards oxidation, which leads to deposits, particularly fouling of injectors and engines.

The “first generation” of biodiesel is typically defined as a mono-alkyl fatty acid ester, which is usually the product of a transesterification reaction of a straight chain alcohol, such as methanol or ethanol, with a fat or oil (triglyceride) to form glycerol (glycerin) and the esters of long chain fatty acids (biodiesel). Most often, methanol is used as a reactant, and the biodiesel is a fatty acid methyl ester (FAME). If ethanol were used it would be a fatty acid ethyl ester (FAEE). As the commercially produced first generation biodiesel is almost exclusively FAME, most of the discussion and analysis in this report refers to FAME. Assuming adequate processing, the feedstock oil determines most of the properties of biodiesel. Biodiesel has virtually no sulfur⁽⁴⁾, nitrogen or aromatic compounds, and contains approximately 11 wt% oxygen. Blends of biodiesel with diesel typically vary from 5 vol% (B5) to 20 vol% (B20) with 100% (B100) biodiesel used in captive fleets in some economies.

The “second generation” and “third generation,” so-called “advanced biofuels,” have a chemistry that is fundamentally different to first generation biodiesel, or FAME. These fuels are often referred to as “renewable diesel.” Although, they have a similar chemical structure to petroleum diesel, they are produced from renewable feedstocks and possess superior performance characteristics, such as very high cetane number and low viscosity. The production processes involve thermochemical conversion, including pyrolysis, gasification and Fischer-Tropsch (FT) processing, and hydrotreating. Advanced biofuels offer several advantages, such as closer matching with current diesel characteristics and fungibility in existing logistics systems. They can also be

² The APEC expert group on new and renewable energy technologies will carry out two projects in 2007 to establish “Guidelines for the Development of Biodiesel Standards in the APEC Region” as well as “Alternative Transport Fuels Policy Options for APEC Economies.”

³ APEC is comprised of: Australia; Brunei Darussalam; Canada; Chile; People's Republic of China; Hong Kong, China; Indonesia; Japan; Republic of Korea; Malaysia; Mexico; New Zealand; Papua New Guinea; Peru; Philippines; Russia; Singapore; Chinese Taipei; Thailand; United States of America (U.S.) and Viet Nam.

⁴ Sulfur can be present from the feedstock oil, mainly originating from fertilizers, but can also be present if sulfuric acid washing takes place and removal is not effective.

produced from a much wider range of raw material, especially waste, and the performance characteristics of the resultant product are virtually feedstock independent.

At the present time, a great deal of second generation biodiesel activity is occurring in Europe (see Figure 1). Although non-esterified second generation biofuels are currently not prevalent in the APEC region, economies such as China, Japan, Australia and New Zealand are already conducting trial biofuel production from wood, grass, algae and other biomass resources, and it is a stated long-term goal of the region to develop low-cost technologies to produce biofuels from non-edible biomass. These technologies are, however, most probably a number of years away from significant market adoption. They involve major investment in high pressure and temperature processes.

As 2nd and 3rd generation biodiesel is largely feedstock independent and will be produced to meet or exceed conventional diesel specifications, the focus of this study is on developing a suitable common performance based standard for 1st generation biodiesel in the APEC region,

Figure 1: Geographic Introduction of Second Generation Biodiesel



Source: Advanced Biofuels Coalition

Asia boasts a wide variety of animal fats and vegetable oils suitable for biodiesel production, including waste cooking oil, palm oil, coconut, jatropha, rapeseed, soy and tallow from many animals. In fact, Malaysia and Indonesia are the world's largest palm oil producers, The Philippines is the world's largest coconut producer and exporter and China is the world's leading producer of rapeseed. Thailand and Viet Nam, being agricultural-based economies, are able to grow a variety of suitable crops. However, Asian APEC economies (such as: Brunei Darussalam; Hong Kong, China; Japan;

Singapore; Korea⁵ and Chinese Taipei) are likely to depend on biofuel imports due to insufficient suitable land to produce both food and biofuels. Hence, the opportunity for a regional standard to facilitate trade in biodiesel amongst the APEC member economies is apparent.

Although Europe is not an APEC economy, it has increasing requirements to reduce CO₂ emissions, coupled with a growing shortage of diesel fuel. Thus, Europe represents a large potential biodiesel export market for APEC producer economies. The current European biodiesel specification, EN 14214, is based on biodiesel produced from rapeseed and combinations of oils that together provide similar characteristics to rapeseed oil, including sunflower, soy and palm. Most of the Asian APEC member economies have derived their biodiesel standards from the European standard, with elements from the U.S. ASTM D6751 biodiesel standard, which was developed to address biodiesel produced predominantly from soy and waste cooking oils.

Due to the APEC region's diversity of feedstocks, both the U.S. ASTM D6751 and the European EN 14214 standards for biodiesel are not suitable without adaptation. The Brazilian biodiesel standard, Agência Nacional de Petróleo (ANP) Act No.42, finalized in 2004, allows for much greater biodiesel feedstock diversity. It should also be noted that all of these standards define biodiesel as mono-alkyl esters of long chain fatty acids derived from vegetable oils, animal fats or biomass, and hence eliminate other sources such as coal slurries, non-esterified oils and partially esterified oils.

Economies in Asia Pacific have different climates, feedstocks and vehicle fleets, which may require a different biodiesel standard from the current U.S. and European standards in order to ensure market acceptance and to maximize the opportunity for regional trade. The standard also needs to take into account existing diesel standards and the type of biodiesel producers, both current and expected, where matters such as size, investment, technology, personnel expertise and quality control approaches are important.

This study compares the current biodiesel standards between APEC member economies, Europe, Brazil and India, and outlines the key biodiesel quality parameters required to ensure the durability and performance of vehicles in the APEC region.

⁵ Based on the APEC accepted nomenclature. In this report 'Korea' refers to 'South Korea.'

4. Background

4.1. History of Biodiesel

4.1.1. Early Developments with Straight Vegetable Oil

The very first diesel engine ran on peanut oil in 1905, but thereafter, the increased cheap supply of crude oil totally replaced the use of vegetable oils. The diesel engine and diesel fuel have been in continuous development over the past 100 years to arrive at the high performance levels of today. The fuel crises of the 1970s and 1980s led to vegetable oils and derivatives thereof being among the materials that were extensively investigated as alternative diesel fuels. Vegetable oils, particularly those low in polyunsaturates, were used successfully in the indirect injection type diesel engines of that time. However, the use of unconverted vegetable oils in direct injection diesel engines, the dominant technology today due to their higher efficiency and lower emissions, leads to a multitude of problems. The problems largely result from incomplete combustion and are characterized by nozzle fouling, engine deposits and ring sticking, due to polymerization or degradation of the vegetable oil and lubricant failure, caused by a combination of lube oil dilution, breakdown and thickening.

Despite these drawbacks, vegetable oils have remained attractive candidates for investigation as alternative diesel fuels because they are renewable resources with relatively high heat content and similar boiling range to mineral diesels. The early 1980s saw research programs in various economies worldwide (including Austria, South Africa and the U.S.) on improving vegetable oil use in diesel engines. This led to the development of esterified seed (vegetable) oils around 1985 in Austria and South Africa. Results showed that esterification increased stability, improved cold flow properties and reduced viscosity. This addressed many of the problems experienced with straight vegetable oils. However, crude prices decreased significantly and interest in the use of biofuels waned.

4.1.2. Esterification from the 1990s

From the 1990s global interest in biofuels reawakened due to geo-political tension and associated increased security of supply considerations, and was further strengthened due to the growing awareness of global warming and in particular, the issuing of the Kyoto Protocol in 1997.

Since the 1990s, the majority of biodiesel manufacture and use has been in Europe. Due to advanced technology diesel engines, European biodiesel developed as a methyl ester.

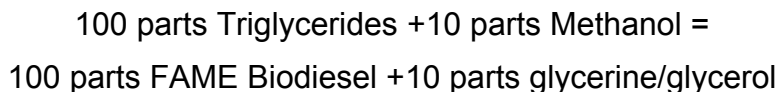
Vegetable oils, new or used, and animal fats, are fatty acid triglycerides. Triglycerides are made up of glycerol and 3 long-chain acids, commonly called fatty acids.

These can be transesterified, involving the replacement of glycerol, a tri-alcohol, with a mono-alcohol, typically methanol. Higher alcohols such as ethanol, and even isopropanol and butanol could be used, but their alternative value in solvents markets,

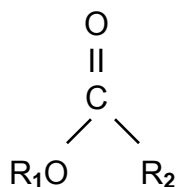
lower reactivity and need to use more per unit of biodiesel, tend to make them less desirable choices. Since water interferes with the transesterification reactions and can result in poor yield of product, the alcohol must be anhydrous.

There are three factors that favor methanol over ethanol, even though methanol is more toxic. The first is the recycling of alcohol during the transesterification process. Methanol is considerably easier to recycle than ethanol, since ethanol forms an azeotrope with water (i.e. boils without change), making it expensive to purify during the recovery step. The second is that methanol is generally much lower priced than ethanol. Thirdly, because ethanol can be used as a beverage, finding sources of pure undenatured industrial ethanol can be difficult. The biodiesel product is termed FAME, where methanol is the alcohol reactant, or FAEE where the alcohol is ethanol. A strong mineral base catalyst such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) is needed to initiate and promote the transesterification reaction because the alcohol is only sparingly soluble in the oil phase, and the reaction occurs via the methoxide ion. After the reaction, the catalyst is neutralized with a mineral or organic acid.

The typical reaction is:



The resultant Biodiesel has a chemical structure as follows, where R_1 is an alcohol and R_2 is the alkyl group:



Fatty acids vary in the length of their carbon atom chain (from 4 to 22) and the number of double bonds they contain. For example, myristic acid (C14:0), palmitic acid (C16:0) and stearic acid (C18:0) contain 14, 16 or 18 carbon atoms in their chain respectively, and no double bonds. Fatty acids are classified according to the number of double bonds they possess. Saturated fats contain no double bond, monounsaturated fats contain one and polyunsaturated fats contain two or more.

To force the reaction equilibrium in the direction of maximum products, one or more parameters of the reaction, such as catalyst, temperature, pressure or the ratio of reactants needs to be changed. In commercial operation, a larger quantity of alcohol (which is recycled) is typically used to drive the reaction to maximum products, thereby minimizing contaminants such as unreacted fatty acids and mono-, di- or triglycerides. Biodiesel feedstocks contain many possible contaminants such as water, free fatty acids, particles and phospholipids. Phospholipids are fat derivatives in which one fatty acid has been replaced by a phosphate group and one of several nitrogen-containing molecules. There is potential for each of these contaminants to impact the quality of the final biodiesel product.

High levels of unreacted oils as well as free and total glycerin in the biodiesel product can cause injector deposits and filter plugging, as well as adversely affect cold flow properties. Other contaminants such as color and odor bodies can reduce public acceptance of the biodiesel if they persist in the final product. The selection of the feedstock oil and pre-treatment processes dictate the production technology that is required and the biodiesel yield that can be expected from that process.

4.1.3. European Leadership

The first FAME standard was introduced in Austria in 1991, based on rapeseed. Other European economies followed, such as Germany, with their standard DIN 51606, which extended possible feedstocks to other plant oils and then to animal fats, by being generic for FAME. After 1997 in the EU, independent national standards were no longer encouraged; the European standard EN 14214 was issued in 2001, largely based on the DIN FAME standard of 1997. The CEN biodiesel working group has adopted a work item to accommodate possible FAEE in the standard.

4.1.4. U.S. Developments

In the U.S., the American Society for Testing and Materials (ASTM) biodiesel task force was formed in 1994 to develop specifications for biodiesel. The philosophy was to develop a stand-alone specification, on the premise that if the biodiesel fuel met the biodiesel specification, it could be blended with diesel. The ASTM D6751 standard assumed the biodiesel would most likely be produced from a mixture of oils and fats, and the standard should be independent of processing methods and set in a similar manner to the way in which diesel specifications are set, i.e. based on properties needed for satisfactory operation of the vehicle and engine, or actual performance-related tests. Most of the feedstocks in use in the U.S. are used cooking oils, soy oil and tallow. The ASTM D6751 standard was developed using the diesel standard as a basis, eliminating specifications not applicable to biodiesel and adding biodiesel specific properties and requirements.

4.1.5. Considerations by OEMs and APEC Needs

The European and U.S. standards were established based on known biodiesel qualities and vehicle fleets in those regions, in addition to philosophies towards development of a biodiesel industry. Several APEC member economies developed similar biodiesel standards, predominantly following the European standard.

Existing diesel fleets have the possibility to reduce emissions, GHGs in particular, by using biodiesel. However, Original Equipment Manufacturers (OEMs) and their industry associations have had problems with varying quality of biodiesel and differences in low sulfur diesels. In particular, they have shown that biodiesel is more susceptible to degradation than conventional diesel. It degrades to form corrosive and deposit forming materials, accelerated by the presence of water, oxygen, heat and certain impurities.

OEMs are conservative regarding fuel changes and variations, as varying fuels increase vehicle compatibility risks and the vehicle technology needs to be more robust and

tolerant with regard to meeting service interval and performance requirements, such as durability and emissions standards. Regrettably, the increasing regulatory and performance constraints on modern diesel engines render them less tolerant to fuel variations. Varying fuels require more extensive and expensive compatibility and durability testing. In particular, the fact that 1st generation biodiesel differs from petroleum diesel is a problem for OEMs; however, this may also represent an opportunity, as biodiesel exhibits certain positive behavior, especially with respect to lubricity.

More than 15 years of experience with extensive biodiesel use has been accumulated, mainly in Europe and U.S. The NREL biodiesel research report (1) of 1997 found more than 350 biodiesel research reports in the U.S. from 1992 to 1997, and the number has increased dramatically over the past 10 years due to increased oil prices and the focus on addressing global warming (Kyoto Protocol).

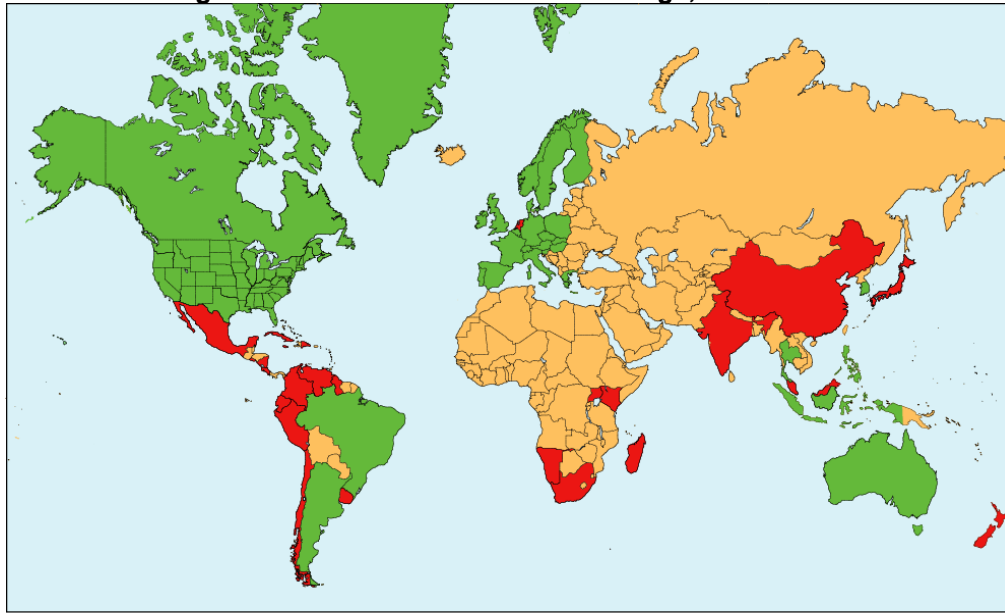
The APEC region has the potential to play a leading role in the development of the most acceptable biodiesel standard(s). This is perhaps appropriate, given that the APEC region is expected to be by far the largest manufacturer and supplier of biodiesel in the next 5 to 10 years⁽⁶⁾. In this regard, the standards of Argentina (2001) and Brazil (2004) may also be informative, as these were more driven from a biodiesel development focus to enable diverse feedstock sources.

4.2. Development of European, U.S. and Brazilian Biodiesel Specifications

The current status of worldwide biodiesel usage is shown in Figure 2 and blending limits in the world are shown in Figure 3.

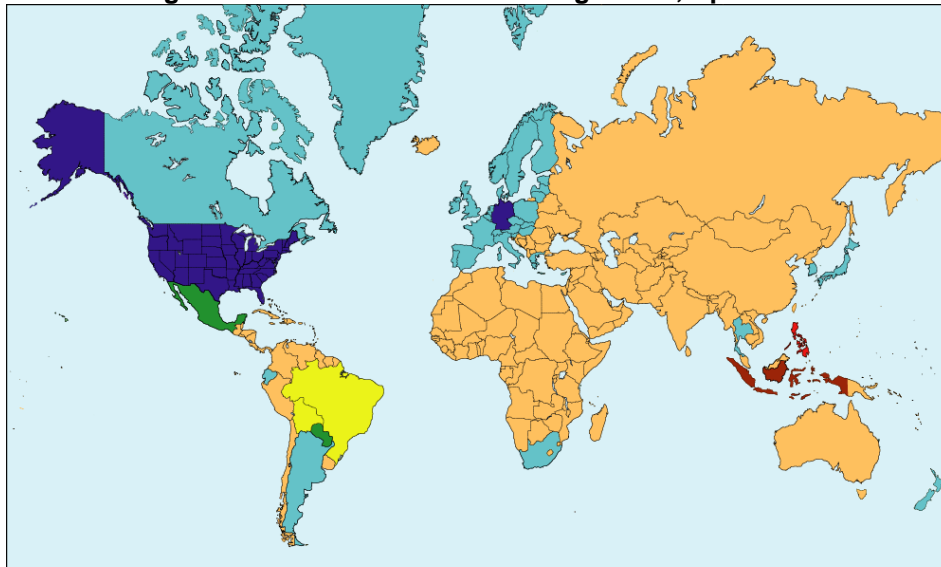
⁶ If all the current world vegetable oil and tallow production was converted to biodiesel, it would make up only approximately 15% of worldwide diesel usage, and approximately 35% thereof would come from APEC economies.

Figure 2: Global View of Biodiesel Usage, March 2007



Source: Global Biofuels Center, 2007

Figure 3: Global Biodiesel Blending Limits, April 2007



Note:

Regions showing pure biodiesel indicates no maximum blend limit set in regional quality specifications

Source: Global Biofuels Center, 2007

4.2.1. Europe

The European Union (EU) represents approximately 70% of worldwide biodiesel production and use, reaching 3.5 trillion liters in 2005 (2) and 6.5 trillion liters capacity in 2006. The EU also has the highest level of diesel penetration in its vehicle fleet and is the leader in advanced diesel technology.

Initial engine tests in the early 1990s were conducted using a rapeseed oil methyl ester (RME). Specification development was highly influenced by the experience of vegetable oil chemistry reflected in European parameters (e.g. iodine value (IV), free glycerin), but parameters of petroleum diesel (e.g. cetane number (CN), Conradson Carbon Residue (CCR)) were incorporated. The need for consistent and well-defined fuel quality was identified as being crucial for obtaining the confidence and support of the automotive industry, particularly the Fuel Injection Equipment (FIE) manufacturers.

Germany followed existing Austrian experience to develop the German DIN (Deutsches Institut für Normung; German Institute for Standards) standards, which are as follows:

- *DIN V 51606 for plant methyl ester of June 1994* – This followed the Austrian example of setting a quality standard for biodiesel as RME under the standard ON C 1190, in 1991. The DIN V 51606 was more flexible by allowing for a broader range of oils of vegetable origin. This fuel standard represented the basis for further increasing numbers of warranties, such as those given by Volkswagen for all their models from construction year 1996 onward; and
- *DIN E 51606 for FAME of September 1997* – This followed the advanced Austrian ON C 1191 for FAME of July 1997 to strengthen the position for issuing further engine warranties and creating customer confidence.

DIN 51606 from September 1977 was issued just before the start of the Comité Européen de Normalisation (the European Committee for Standardization or CEN) process, which was triggered by Mandate 245, given by the European Commission to CEN, which required that any further independent national initiatives should cease. CEN is a non-profit making technical organization set up under Belgian law, and contributes to the objectives of the EU and EFTA economies with voluntary technical standards designed to promote free trade, worker and consumer safety, and optimization of regional networks, environmental protection programs and research and development. The CEN mandate provided for the development of the following standards:

- biodiesel as pure diesel engine fuel (100%);
- biodiesel as an extender (blend component) to diesel fuel, according to EN 590 diesel specification; and
- pure biodiesel or biodiesel as an extender to mineral oil products, for the production of heat in particular.

Stakeholders and specialists participated actively with members present in all working groups, being able to contribute based on the already accumulated experience in the national standards developments. Among those represented were engine and equipment companies, oil companies, biodiesel producers and research institutions. This led to the European standard, EN 14214, in May 2001. This standard's objective

was to make biodiesel a recognized product throughout Europe, which could be made available to all European citizens by free trade and without national restrictions. It also provided guarantees to consumers, a basis for vehicle manufacturers and a requirement for biodiesel producers.

A consequence of the standard was that an essential contribution to the accomplishment of the EU common aim of preserving the environment, securing energy supply and preserving employment in the agricultural sector was made.

The EU as an entity does not have biodiesel waivers, but they can exist individually on Member State levels. Biodiesel can be blended up to 5 vol% in diesel, but the blend needs to meet the same standard (EN 590) as 100% petroleum diesel. Labeling is required for blends above 5 vol%. Certain OEMs and FIE manufacturers have expressed concern regarding the use of higher blends (greater than 5 vol%) in the existing fleet because of issues concerning vehicle compatibility and the potential for increased emissions (3). Given the increased biofuels target for 2010, the European Automobile Manufacturers Association (ACEA) is pressing for a B10 specification to address these concerns.

Blends higher than 5 vol% have generally been used in captive fleets on the basis of national waivers. B100 is however used as neat fuel and is sold in Germany, as a special labeled product, at some 1,700 retail filling stations (approximately 10% of retail sites). The owners of the majority (1,300) of these filling stations have pledged to comply with the standards and requirements of the AGQM⁽⁷⁾ (Arbeitsgemeinschaft umfasst Biodieselerhersteller und Vermarkter, or Biodiesel Quality Management Association), which states that only biodiesel based on RME will be offered for sale at filling stations. This is due to the fact that several FIE and vehicle manufacturers only approve RME or equivalent for use in their vehicles. To comply with these specifications, the blending of biodiesel derived from other feedstocks has to approach the characteristics of rapeseed oil. For example, a typical feedstock blend complying with the European standard is 70% rapeseed, 20% soy and 10% palm oil. In Germany, approximately 33% of biodiesel is sold as B5 at filling stations and 30% is sold as B100 at filling stations. This is roughly equally used by private motorists and fleets. The remaining 37% of biodiesel is sold wholesale to commercial fleets⁽⁸⁾.

4.2.2. U.S.

In the U.S., biodiesel production has been increasing rapidly reaching 205 million liters in 2005 and almost double that in 2006, with current planned capacity of 4,500 million liters, mainly from soy oil (2). The ASTM issued Specification D6751 is for all B100 biodiesel fuel bought and sold in the U.S. The ASTM Committee D2 approved it in December 2001 after more than 7 years of work by the ASTM Biodiesel Task Force. Represented on this committee were 290 organizations from 17 economies. The development and enhancement of the D6751 standard has been essential to the

⁷ www.agqm-biodiesel.de.

⁸ www.ufop.de/downloads/Vehicles_Biodiesel.pdf.

commercialization of biodiesel in the U.S. ASTM D6751 focuses on B100. Currently, if B100 meets D6751 and petroleum diesel fuel meets ASTM D975, there is no separate set of specifications for the blend. However, the ASTM is discussing the incorporation of up to a B5 blend into the D975 specification and the establishment of a separate specification for B6 to B20 blends.

In the U.S., most of the experience with biodiesel has been with B20, with market share of diesel/biodiesel blends being approximately 75% B20, 24% B5/B2 and 1% B100 (4). The use of B20 has been the subject of quality and stability surveys annually (3), and few operational problems are experienced. Most of the established problems are usually caused by biodiesel not meeting requirements of D6751 (high glyceride or soap content) or other issues (cleaning effect of biodiesel, poor cold flow, microbial growth, improper blending). Nevertheless, there is no certainty that a compliant biodiesel added at 20 vol% to on-specification (mineral) diesel will result in an on-specification blend, in regard to the diesel standard. The ASTM Working Group WK 7852 has been working towards producing a B20 standard. Since B20 samples show high levels of oxidation, a critical issue in this process is the establishment of a stability specification in ASTM D6751 and perhaps in the future B20 standard.

4.2.3. Brazil

Brazil has developed a national biodiesel program as a way of stimulating agriculture and employment, by mandating 2 vol% in 2008 and 5 vol% in 2013. This equates to 900 million liters in 2008; installed capacity has almost reached that target. In December 2004, Brazil issued their national regulated specification ANP 42/04, which differs from EN 14214 and ASTM D6751. They focused on performance based parameters in the specification allowing multiple feedstocks. They similarly state (5) that biodiesel is a fatty acid methyl (or ethyl) ester, produced by transesterification reaction of vegetable oils with methanol (or ethanol). In July 2006, a B2 specification was implemented.

To be more multi-feedstock enabling, Brazil classified properties related to the “quality of the process” versus those related to the “nature of the raw materials” (the feedstock) (see Table 1) with the aim of complying with end market use requirements. In addition, no limits are set for properties where either the analytical method (for multi-feedstock) or the actual limit is still undefined due to the diversity of raw materials, and where no performance issues are anticipated. These properties are only required to be reported quarterly. The intention is to use this database of reported values as a basis for setting further specifications if required, perhaps even with blends of biodiesel from different feedstocks.

For logistics and distribution, Brazil requires that the blend (Bx) must comply with the diesel specification, and does not limit, nor require the testing of biodiesel alone. Such non-limit tests can be conducted to assist blend simulations (modeling calculations) and evaluation, so as to facilitate blend compliance.

The Brazilian philosophy to biodiesel specifications could be described as recognizing biodiesel as a blend component, for which the properties may not comply with the end market specification, but can be used via blend synergy to formulate a diesel that

does comply with performance requirements. The Brazilians conducted extensive studies to establish that their specification did not limit the diversity of biodiesel sources. Interestingly, this is the same approach taken in a conventional oil refinery, where marketable diesel is blended from a range of diesel blend components that individually do not typically comply with the final product specification.

The Brazilians have conducted extensive tests to establish how the biodiesel components blend, whether linear (such as density), or according to a relatively simple polynomial blend model (viscosity), or a more complex blend model that may need empirical testing (such as iodine number). They have been promoting their approach to establish a single uniform international specification. They recognize that this will require more work on defining pertinent qualities of feedstocks and development of analytical methods.

Table 1: Brazilian Approach to Biodiesel Specification Parameters

Process Dependent	Applied by Testing
Visual appearance	Every batch
Water	Every batch
Total contamination	Report by testing 3 monthly
Acidity; Copper corrosion	Every batch
Flash point; methanol content	Every batch
Free and Total glycerol	Every batch
Ester content; mono-, di-, triglycerides	Report by testing 3 monthly
Na & K	Every batch
Ca & Mg; Phosphorous	Report by testing 3 monthly
Carbon residue; sulfated ash	Every batch
Raw Material Dependent	
Density; viscosity; CFPP	No limits for biodiesel alone
90% distillation	Every batch
Cetane number	Report by testing 3 monthly
Iodine number	Report by testing 3 monthly
Oxidation stability	Every batch
linolenic acid methyl ester content polyunsaturated methyl esters	No limits for Brazil and ASTM; only EN
Both Process & Raw Material Dependent	
Sulfur	Report by testing 3 monthly

Source: Parente et al. (5)

4.3. Second Generation Biodiesel

These biodiesels have a chemical structure that is more similar to conventional diesel, but are produced from renewable sources. There are technologies for second generation biodiesel under development, but not yet commercially significant, including:

1. Hydrogenation of fatty acids (vegetable oils), either in dedicated units⁽⁹⁾ or combined into conventional diesel hydrotreaters⁽¹⁰⁾. This primarily removes unsaturated (olefinic) bonds and oxygenates, with waste co-products being mainly water and carbon dioxide (CO₂). This type of process would compete with the current conventional biodiesel esterification process as the same feedstocks are used. In addition, it should be noted that first generation biodiesel could also be hydro-processed, but this would significantly increase processing costs;
2. Hydrogenation of bio-oil (a bio syncrude) produced by pyrolysis of biomass⁽¹¹⁾ (heating and driving off volatiles, that are collected as the syncrude oil);
3. Conversion of biomass to syngas⁽¹²⁾ by gasification and then Fischer Tropsch (FT) reaction to a synthetic oil⁽¹³⁾. This is termed BTL (biomass to liquids), and the properties do not differ materially from GTL (gas to liquids) or CTL (coal to liquids) products, since the route is via syngas. The properties of this syncrude and the derived diesel, jet fuel, gasoline, naphtha and LPG (liquefied petroleum gas) are largely covered by the conventional crude oil derived specifications, sometimes with additions such as control of oxygenated co-products and modifications such as lower density and viscosity.

Options 2 and 3 above would compete as they would use similar biomass feedstocks or similar land being used to grow the feedstocks, and would further compete with alcohol type second generation technology such as enzymatic hydrolysis and fermentation. These processes could also be complimentary by being staged and optimized to the bio-feed properties. For instance, pyrolysis to drive off bio-oil as volatiles can be followed by gasification of the devolatilized biomass. Pyrolysis and/or gasification could also address the cake co-product from which the vegetable oil is expelled, or the biomass part from oil seed trees or the bagasse from sugar cane.

All the above bio-derived fuels with distillate fraction boiling in the diesel range have favorable properties such as very low-levels of aromatics, sulfur and low density, and as such, have been widely acknowledged by engine and fuel system component manufacturers as leading diesel fuels. In addition, they can be included into conventional refining processes and logistic systems, and appear to be quite well suited to conventional crude oil type diesel specifications. However this needs to be confirmed, and the ASTM has already set up a task force to address the question of where such renewable hydrocarbon fuels fit into the specifications.

The production facilities for second generation biodiesel require hydrogen in large quantities, as bio-oils have relatively high oxygen contents. They also employ high pressures and high temperatures, and are thus likely to be large scale and integrated with existing oil refineries (with about a minimum of 20,000 b/d or 1 billion liters/year).

⁹ UOP and ENI, Neste and Total as technology developers.

¹⁰ IFP, Petrobras, Conoco-Phillips as technology developers.

¹¹ Lurgi as a technology developer.

¹² Carbon monoxide and hydrogen.

¹³ Shell/Choren and Sasol/Chevron as technology developers.

The associated investment would typically be in excess of US\$1 billion, and in such cases, the developers would need to address market fit and compliance as part of bankability.

In contrast, esterification and transesterification is carried out at low temperatures on a smaller scale (typically in the range 10 -100 million liters/year or 200– 2,000 b/d), and with significantly less investment (in the range of US\$1 million – US\$50 million). The properties of the product, as well as the effect of the feedstock and process on the product differ significantly from those of conventional diesel and therefore this 1st generation biodiesel requires a separate “biodiesel” standard. Therefore, the APEC biodiesel standard that is being developed focuses on 1st generation biodiesel at this time.

4.4. Biodiesel Compared to Petroleum Diesel

It is useful to identify differences and similarities between biodiesel and diesel. While biodiesel is generally considered to be a substitute for all or part of diesel fuel, there are some key differences that need to be considered when examining pure biodiesel replacing diesel or when it is blended with diesel. Key differences, explored later in more detail, are:

- Biodiesel is a combination of a small range of molecules, typically esters of fatty acids of C₁₂, C₁₄, C₁₆, C₁₈ and C₂₂, whereas diesel is a complex mixture of a broad range of hydrocarbons from C₁₂ to C₂₅, consisting of paraffins, naphthenes and aromatics, as well as a range of nitrogen and sulfur containing organic compounds;
- Diesel is produced largely by distillation of a broad cut of petroleum, to separate the lighter (lower flash point gasoline and kerosene) and heavier (typically waxy, fuel oils and heavier marine distillates) components, whereas biodiesel is produced by a chemical reaction followed by physical separation. Biodiesel thus may contain some heavy materials or materials that are subject to thermal decomposition when exposed to heat occurring in an engine;
- Biodiesel may, and often does, contain a high level of unsaturated (olefinic) carbon bonds. Normal crude oil contains few olefins and refinery hydrotreatment, meant to reduce sulfur, further lowers this. Olefins are unstable and may contribute to deposits and higher rates of degradation; and
- Biodiesel contains oxygen, as the ester, which fundamentally affects its performance properties. It reduces energy content and makes the biodiesel polar, through the hydroxyl (-OH) hydrogen bond. The polarity gives it properties of solvency, detergency, wet-ability (sticking to metals as a lubricant) and conductivity.

4.5. Sources of Feedstock in APEC Economies

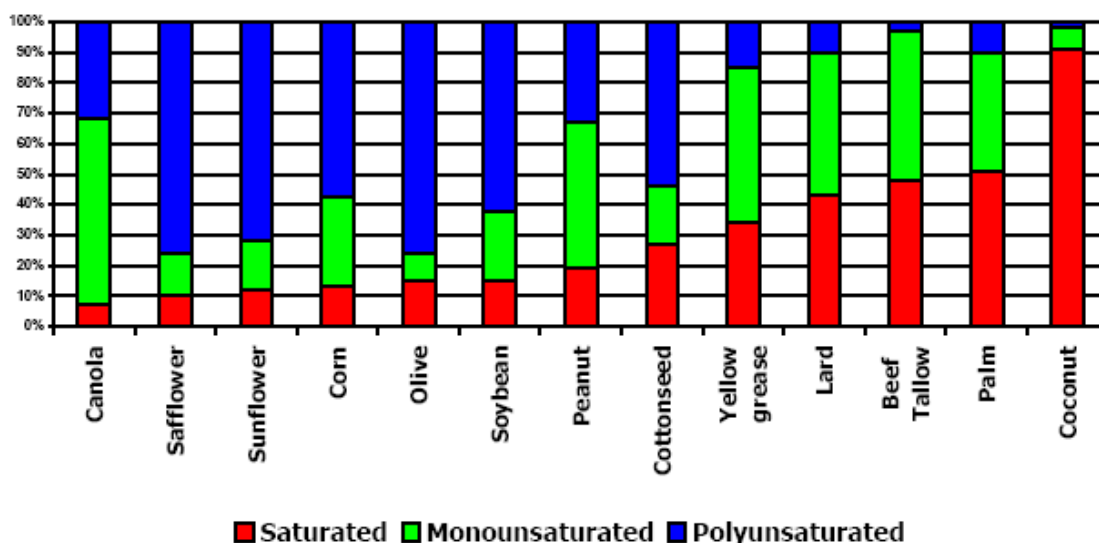
Biodiesel can be derived from many sources that contain fat or oil. Examples of the most common types of biodiesel used in some economies are (6):

- CME = Coconut Methyl Ester (The Philippines) from coconut oil,

- TME = Tallow Methyl Ester (Australia) from sheep (mutton) and beef bovine fats,
- PME = Palm Methyl Ester (Malaysia) from palm oil,
- JME = Jatropha Methyl Ester (India) from jatropha oil,
- RME = Rapeseed Methyl Ester (Europe) from rapeseed oil,
- SME = Soy Methyl Ester (USA, Argentina) from soy oil, and
- FAME = Fatty Acid Methyl Esters is a generic term for biodiesel. It represents all alternative diesels produced from vegetable oils or animal fats, reacted with methanol.

What makes each of these feedstocks different from the others is that they are made of different proportions of saturated, monounsaturated and polyunsaturated fatty acids (Figure 4), with differing chain lengths or number of carbon atoms, and degree of branching of these chains. A “perfect” biodiesel, as regards stability and cetane number, would be made only from monounsaturated fatty acids (see Table 2).

Figure 4: Composition of Various Biodiesel Feedstock



Source: Tyson et al. (7)

Table 2: Fuel Properties as a Function of Fuel Composition in Diesel Engines

	Saturated	Monounsaturated	Polyunsaturated
Fatty acid	12:0, 14:0, 16:0, 18:0, 20:0, 22:0	16:1, 18:1, 20:1, 22:1	18:2, 18:3
Cetane Number	High	Medium	Low
Cloud Point /CFPP	High	Medium	Low
Stability	High	Medium	Low

Note:

The first number of the combination shows the number of carbons in the fatty acid chain; the second number is the level of saturation or unsaturation – 0 for saturated, 1 for monounsaturated, and 2 or 3 for polyunsaturated.

Source: Tyson et al. (7)

4.6. Diesel Vehicles in APEC Economies

Generally, most vehicles that can operate on diesel with 500 parts per million (ppm) sulfur content or lower, are compatible with blends of suitable biodiesel with diesel at low-levels, ie. up to B5. Within the APEC region, Indonesia, Malaysia, Peru and Russia are supplying diesel with sulfur levels higher than 500 ppm. Russia supplies both high and low sulfur diesel and the high sulfur diesel is intended for older vehicles. However, all these economies are setting road maps for improved fuel quality with lower sulfur diesel by 2011 or earlier.

This indicates that B5 should be allowed in general as an alternative to diesel and that labeling of B5 blends (as is the case in the EU) with the most sensitive vehicle parc is not likely to be necessary, subject to the quality of the biodiesel and the blend interactions with the particular diesel being typical of existing experience.

Most of the APEC economies (with the exception of the U.S. Canada and Japan) have a greater proportion of heavy-duty diesel vehicles and older parcs with older technology compared to the EU, so a higher proportion of the diesel engines in APEC region should be more tolerant of biodiesel properties (see Appendix section 10.4). The heavy-duty vehicles that are dominant consume more fuel per kilometer than light-duty diesel engines and also typically operate much more, covering higher mileages. Hence, by far the greatest proportion of biodiesel is used in heavy-duty diesel vehicles. Blending a limited quantity of palm and coconut oils into diesel without transesterification to alkyl esters could be considered if the fuel was only to be used in heavy duty applications in Asian APEC economies. However, developing a biodiesel standard for light-duty high speed applications ensures a fuel that is compatible with both light and heavy-duty diesel vehicles and fulfils the primary objective of facilitating maximum trade in biodiesel between APEC member economies.

4.7. Specification, Quality Control and Monitoring Approaches

4.7.1. Market Experience and Quality Levels

Experience has shown that biodiesel market problems often have less to do with the standards, and more with poor manufacturing practices and quality control resulting in biodiesel not complying to the standards in place. In 2006 in the U.S. a survey (1) found that 50% of samples failed to meet the ASTM specification. In particular, glycerin levels were too high for 30% of the samples, and 20% failed on sodium (Na) and potassium (K) levels. These failures resulted from production problems. The product quality failures contributed to filter blockage and degradation with resultant high acid values and deposits. For this reason, effort should be focused on ensuring a standard that is workable for biodiesel producers, and this needs to be backed by quality control and an assurance program that supports high levels of compliance. Those violating the standard should be prosecuted

4.7.2. Consumer Needs and Market Impacts

Consumers want assurance that the biodiesel or biodiesel blend they purchase will result in reliable performance from their vehicles, so the specification must address marketability. If a specific market problem is encountered because of a specification failure, rapid problem analysis and corrective action must follow. This could address the biodiesel specification or the application, such as changing vehicle parameters, e.g., servicing intervals and procedures or limiting the use of the biodiesel. A proper reporting and monitoring program is therefore key to national biodiesel programs. Biodiesel use is still limited, compared to mineral diesel, and is growing in a more stepwise manner than would occur with major refinery changes, so the market experience lessons tend to be learned in a stepwise manner. This lowers risk and enables a progressive approach to be considered. Such an approach was followed in Australia, where Environment Australia, representing the government view, stated: "The suite of parameters proposed for implementation on (Sept. 18, 2003) will ensure a quality biodiesel product enters the Australian market. As the biodiesel industry will need time to access quality analysis infrastructure, establish analytical techniques and gain accreditation for some test methodologies, it is proposed that there will be a progressive introduction of further parameters over time. This approach has previously been successfully adopted for automotive diesel and unleaded (gasoline)."

In developing an APEC biodiesel standard, a similar approach may be followed, including:

- agreeing on a standard sooner, perhaps even without all the answers,
- making sure sufficient certified testing facilities are available,
- providing good quality assurance programs,
- providing good field experience monitoring,
- imposing stiff penalties for violators, and
- enabling a process for adjustments and advancements when necessary.

4.7.3. Quality Program Needed

A successful biodiesel trade amongst APEC member economies requires a quality assurance program to complement the biodiesel quality standard. This has been demonstrated by the German AGQM and U.S. BQ-9000 quality programs.⁽¹⁴⁾ These assurance and monitoring steps are more suited to national programs, linked to biodiesel producer voluntary associations and/or government support and regulatory schemes. For further insights, a brief description of the U.S. and German quality programs is given in Appendix 10.5.

¹⁴ In Appendix 10.5.1, some aspects and lessons from the U.S. BQ-9000 quality program are included.

5. Biodiesel Properties

The 1st generation fatty acid alkyl ester biodiesel differs from diesel in terms of chemical composition and physical properties, such as density, viscosity and cold flow properties. Many of the differences are related to oxygen content and the polar nature of biodiesel compared to diesel. Slight polarity is a characteristic of esters compared to conventional hydrocarbons, mainly due to the presence of the hydroxyl ion (-OH) in the molecule.

5.1. Production Effects

Ultimately, the quality of biodiesel is a function of:

- feedstock properties,
- process, including the control of the production process (the expression “built in quality” applies)

Biodiesel can be produced using a batch or continuous process, with in-line analyzers or sample testing throughout the process to control the quality of the product as it is produced. The final product quality should also be tested in tankage to ensure that it meets the specification before it is despatched.

The specification, apart from setting the desired performance requirements in the product, must also consider and control the feedstocks and process effects. It is thus vital to any standard to understand the process impacts and feedstock effects.

5.1.1. Feedstock

The type of triglyceride in the feed varies primarily by chain length, branching and degree of saturation, and is divided into three groups, as shown in table 3. The properties of these fatty acids influence the final biodiesel product properties and quality.

The three component fatty acids and the biodiesel product can all be the same or all be different. The higher the level of saturation, such as palm oil and animal fat, the better the stability, but the poorer the low temperature handling.

Table 3: Saturation Levels of Differing Feedstocks

	Saturated g/100g	Mono Unsaturated g/100g	Poly Unsaturated g/100g	Iodine Value
Palm oil	85.2	6.6	1.7	54
Coconut oil	45.3	41.6	8.3	10
Animal fat	40.8	43.8	9.6	40–50
Rapeseed oil	5.3	64.3	24.8	98
Soy oil	14.5	23.2	56.5	130
Sunflower oil	11.9	20.2	63.0	135

Source: Terry de Winne, "Biofuels for Sustainable Transport" (<http://www.biofuels.fsnet.co.uk>) and the National Biodiesel Board

The transesterification process works on all kinds of triglycerides, but some characteristics of the ester are directly linked to the properties of the feedstock oil, namely:

- Density; viscosity; cold filter plugging point (CFPP); 90% distillation
 - These are determined by the molecular weight (number of C-atoms) – being lower for less carbon atoms; branching – lower for more branched molecules; and degree of saturation – unsaturation leads to better cold temperature properties.
- Cetane number
 - Longer and straighter chains generally impart high cetane, but this falls as the degree of unsaturation rises.
- Iodine number
 - This is a measure of unsaturation or olefin content, as iodine reacts with the unsaturated bonds. A higher number would imply poorer stability.
- Oxidation stability
 - This is better for more saturated (less olefinic) triglycerides. As the number of double bonds rises the propensity to oxidize in the presence of air rises, leading to instability.
- The purity and chemical composition of the feedstock
 - also affects the yield of the final products.

Many of the general properties of biodiesel are therefore directly dependent on the fatty acid ester composition. For example, the U.S. mainly uses soy with a relatively high degree of unsaturation, hence relatively poor oxidation stability but good cold flow properties. Rapeseed oil, used mainly in the EU is an excellent raw material for the manufacture of biodiesel, as it enables a CFPP of -10°C to -12°C to be achieved, and has good oxidation stability with low antioxidant additive treats. Malaysia uses palm oil feedstock with a high level of saturation, which imparts good oxidative stability but poorer cold flow properties. The Philippines uses coconut oil feedstock, also with a high degree of saturation, but with mostly C_{14} molecules and biodiesel with low CFPP of -5 °C. The use of high, unsaturated triglyceride oils such as sunflower oil as a fuel may cause significant performance problems including formation of deposits and

thickening. These problems can be addressed to some extent by anti-oxidants, typically at higher dosage rates.

Generally, the higher content of FFA, water, phosphorus, sulfur and other contaminants in the feedstock, the more expensive or difficult the biodiesel production process becomes. It is therefore critical to properly store the feedstock oil, and when stocks are built in the oil seed harvesting season for later use, a specific anti-oxidant stabilizer may be required.

5.1.1.1. Free Fatty Acid

The presence of free fatty acids (FFA) leads to the use of an increased amount of catalyst. This results in higher salt and water concentrations in the crude glycerin because an increase in catalyst increases the amount of acid needed to neutralize the catalyst. During transesterification, FFA combines with the NaOH or KOH catalyst to form soap. Soap formation causes foaming and emulsions, resulting in more difficult phase separation, and leading to greater contamination of the biodiesel.

To prevent these problems, the best results are achieved by first using a standard process in vegetable oil refining to chemically neutralize oil to a low FFA content, typically below 0.5%.

5.1.1.2. Insolubles

The content of insolubles results in a tendency to form sediment, and hence should be as low as possible in the feed oil, as these substances typically pass through the process and are found in the final biodiesel product, causing premature wear of the fuel injector systems. Sediment may consist of suspended rust and dirt particles, but it may also originate from the fuel as insoluble compounds formed during fuel oxidation. Shipping could increase levels of sediment. Changing from petroleum diesel fuel to biodiesel may initially cause an increase in contamination as the more polar biodiesel has greater solvency and tends to loosen sediments. This would increase fuel filter blockage during the changeover period. The state and type of distribution systems, such as the presence of filters, determines whether this parameter (total contamination specification, IP 440) should be included in the biodiesel specification. It is normally not grounds for refusing to accept product at the exchange of product level, typical of bulk supply, as subsequent settling or filtration can ensure compliance. This may be linked to a cost penalty claim from the purchaser to the supplier/manufacturer. For this reason, this is more a market than a production limit, and biodiesel traded between economies should meet the same standard as for conventional diesel in that market. Most economies that specify total insolubles have set the maximum limit in biodiesel at 24 ppm, as shown below. The European CEN is currently discussing a lower specification limit and more precise test method.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Total contamination, ppm	24	Report	–	24	24	24	–	24	24	–	–	24	–

5.1.1.3. Iodine Value

The traditional measure of the degree of double bonds available for polymerization (thickening and deposit formation) is the Iodine Value (IV). This can be determined by adding iodine to the fat or oil, and the amount of iodine in grams absorbed per 100 ml of oil is reported as the IV. The higher the IV, the more unsaturated the oil and the greater its potential to polymerize at the high temperatures of the diesel engine. Polymers lead to clogging of filters, deposits inside fuel injection equipment, nozzle coking and seizure.

Some oils have a low IV and may even be suitable as a diesel component without any processing. This study limits scope to esters, and the majority of vegetable and animal oils have an IV that may cause problems if used as a neat fuel. Table 4 lists various oils and some of their properties.

Table 4: Oils and Their Melting Point and Iodine Value

	Approx. melting point °C	Approximate Iodine Value
Coconut	25	6 – 12
Mutton/beef tallow	42	35 – 50
Palm	35	35 – 61
Castor	-18	82 – 88
Peanut	3	80 – 106
Rapeseed	-10	94 – 120
Cotton seed	-1	90 – 119
Sunflower	-17	119 – 138
Soybean	-16	117 – 143

Source: Terry de Winne, "Biofuels for Sustainable Transport" (<http://www.biofuels.fsnet.co.uk>) and National Biodiesel Board

The use of methanol versus ethanol as a catalyst also affects the IV of the resultant biodiesel. The IV can be reduced by hydrogenation of the oil, with the hydrogen breaking the double bond and converting the fat or oil into a more saturated oil, which reduces the tendency of the oil to polymerize. However, this process also increases the pour (and melting) point of the oil. As can be seen from Table 4, coconut oil has an IV that is low enough to be considered for use without any potential stability problems in certain unmodified diesel engines. However, with a melting point of 25°C, the use of coconut oil in cooler areas would obviously lead to fuel blockage problems, so transesterification is necessary to split the molecule into three smaller molecules, and hence reduce the pour point.

Those economies with an Iodine Number specification are shown in the table below.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Maximum Iodine Number	-	Report	-	120	120	Report	115	120	120-125	-	-	120	-

The European CEN is considering increasing its Iodine number limit to 130. Arguments have also been made to remove the iodine number specification in favour of direct oxidation stability testing, but defining the stability test method is important. Additives can be used to control oxidation stability but they typically cannot control the sludge forming tendency of the biodiesel.

5.1.1.4. Phosphorus

Phosphorus in oil is present as part of the complex lecithin molecules, often derived from fertilizers, which are good emulsifiers. In practice, the emulsions will be present as an in-between layer in the separator after transesterification. Regular removal by pumping the interface is necessary to prevent further disruption of the separation process. This layer is taken to the glycerol treatment, where contact with acid will split the emulsion into ester/fatty acid and glycerol. Therefore, a high phosphorous content results in a loss of ester, reducing biodiesel yield and economics. Literature (8) indicates that a 3 ppm to 4 ppm level of phosphorous is acceptable and that from 5 ppm to 6 ppm or more, emulsion problems may occur.

In addition to creating process problems, an increase in phosphorus content increases the likelihood of phosphorus in the biodiesel product. Phosphorus poisons the exhaust after-treatment catalyst devices.

The tabulated comparison shows that a maximum of 10 ppm is common for final biodiesel.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Phosphorus, ppm, max	10	Report	-	10	10	10	10	10	10	10	10	10	10

Brazil focuses on the blended diesel, and hence only requires the phosphorus content in biodiesel to be reported. Their view is that there is little or no difference to after-treatment devices if B10 with the biodiesel containing 100 ppm phosphorus and the conventional diesel containing no phosphorus is used.

It should be noted that the CEN experts are discussing the reduction of phosphorus

content in the EN 14214 European specification to 2 ppm in order to facilitate the long-term emissions conformity of the latest diesel vehicles.

5.1.1.5. Stability and Deposits

Biodiesels are more susceptible to oxidation than petroleum diesels and tend to produce gums and lacquers, creating potential for significant increases in deposits. Modern diesel direct injection engines have smaller injectors operating at higher pressures and hence are more susceptible to fouling and malfunction(12). Specific oxidation inhibitors are therefore required, and the European industry has moved to antioxidant treatment as part of the biodiesel manufacturing process (13). A recent study showed that the natural detergency of biodiesel, coupled with the absence of high boiling point materials resulted in less injector deposits (14). Biodiesel and biodiesel blends respond well to conventional diesel dispersant additives to control deposits (15).

Biodiesel degrades about four times faster than petroleum diesel. Within 28 days, pure biodiesel degrades 85% to 88% in water. Blending biodiesel with diesel fuel tends to accelerate its biodegradability. For example, blends of 20 vol% biodiesel and 80 vol% diesel fuel degrade twice as fast as diesel alone. This can be addressed by antioxidants, although more development is still occurring, particularly for biodiesels produced from different feedstock (16).

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Oxidation stability at 110°C, hr, min	6	6	6	6	6	6	-	*	6	-	6	6	3

Note: * Based on mutual agreement between parties concerned.

As shown in the table above, there has been a general agreement in the national specifications of various economies that a minimum oxidation stability of 6 hours is required using the Rancimat method. However, Japan tested biodiesel meeting the European specification and used in a B5 blend over a period of two years, and found material compatibility failure (corrosion in tern sheet) and fuel tank test failure (by corrosion and melting plating in lead-tin alloy coated and electrolytic zinc-coated steel sheets). About 80% of the fuel tanks found in Asia are metal, compared to less than 10% in the EU, which may explain some of these failure results. As a result, Japan is recommending an oxidation stability limit of 10 hours minimum. The CEN in Europe is discussing increasing the limit to 8 hours or more due to potential vehicle problems in the market, and also reconsidering the acid number specification, as well as introducing limits for trace metals such as Cu and Zn found in fuels and shown to have a marked effect on increasing the oxidation tendency of the fuel.

Peroxides (hydroperoxides) are reactive oxidizing agents formed during the first steps of fuel oxidation, or primary oxidation products. Although not a biodiesel specification,

as peroxides rapidly react further to form acids and gums (specification), Peroxide Value is useful in determining the actual progress of biodiesel degradation.

The maximum acid value and in particular, the maximum linolenic acid methyl ester content of biodiesel is specified as follows:

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Maximum Acid Value, mg KOH/g, max	0.8	0.8	0.8	0.5	0.5	0.5	0.8	0.5	0.5	0.5	0.5	0.5	0.5
Linoleic Acid methyl ester, wt% max	-	-	-	12	12	-	-	12	12	-	-	12	-

The acid number specification protects against injection system degradation. The ASTM is considering decreasing this limit to 0.3 mg KOH/g, and the Japanese Automotive Association (JAMA) has proposed a Total Acid Number (TAN) specification of 0.13 mg KOH/g maximum as a stability criterion.

The Linolenic acid methyl ester specification of 12.0 % maximum protects against extremely unstable and polymerising oils. The European CEN is discussing a specification to limit polyunsaturates with more than 4 double bonds to a maximum of 1% to further address biodiesel instability, but a suitable test method has not yet been developed.

To control deposits further, particularly important when used cooking oils are the feedstock, a maximum residue, the CCR test, as developed for petroleum diesel and other petroleum products, has been utilized successfully for biodiesel. In fact, it is more important for biodiesel as it indicates impurities, such as free fatty acids, glycerides, soaps, insolubles, polymers, additives like pour point depressants and content of higher unsaturated fatty acids.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
CCR, 100%, wt%, max	0.05	-	-	-	-	0.05	0.05	-	0.05	0.05	0.1	-	0.05
10%, wt%, max	0.3	0.1	0.3	0.3	0.3	-	0.3	0.3	0.3	-	-	0.3	-

This test indicates composition and impurities that cause harmful deposits in injection systems. Different economies typically follow either the EU standard of measuring Conradson carbon residue on the 10% distillation residue, versus the ASTM philosophy of using the full biodiesel sample. The question of sample preparation (original sample or 10% distillation residue) should be reconsidered. To get a 10% distillation residue, biodiesel has to be distilled under high vacuum. The instrumentation for such

distillation is costly and only few laboratories are able to conduct this test. It is extremely time consuming and the results have poor reproducibility. From a scientific standpoint it does not seem to make any sense to produce a distillation residue from biodiesel, which in contrast to diesel, has almost the same boiling point throughout the product. The CEN is currently discussing changing their standard to use the 100% sample for testing, with the limit value adjusted accordingly. Experience with CCR of the original sample has shown that this method gives excellent data for quality control of biodiesel (17).

5.1.1.6. Sulfur

Sulfur present in feedstock will typically end up in the biodiesel. Vegetable oil feedstocks typically have very low-levels of sulfur of less than 100 ppm and often below 10 ppm, so this only becomes an issue for certain feedstocks when product sulfur levels below 50 ppm or 10 ppm are specified. When very low sulfur limits apply, feedstocks with high sulfur need to be either blended with a low sulfur feedstock in order to reduce the amount of sulfur in the biodiesel product or the resultant biodiesel needs to be blended with ultra low sulfur petroleum diesel, such that the blend complies to specification. The diesel sulfur specification is ultimately based on emission requirements and/or vehicle technology durability requirements. The sulfur levels in fuels are frequently regulated on a national or regional basis in order to facilitate emissions conformity of vehicles with the latest technology and also to improve the emissions performance of existing fleets. Biodiesel producers may need to pay special attention to the sulfur parameter in their production processes in the future.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Sulfur, ppm, max	10	Report	50	10	10	50	100	10	50/10	500	10	10	15/500

5.1.1.7. Water

The presence of water in feedstocks or the process creates soap and the problems detailed in 5.1.1.1 above. Water is formed when producing the methoxide catalyst by reacting methanol with sodium or potassium hydroxide.

Water in biodiesel can be present in two forms: dissolved water or suspended water droplets. While biodiesel is generally considered to be insoluble in water, its polarity tends to make it dissolve more easily than diesel fuel. Biodiesel can contain as much as 1,500 ppm of dissolved water while diesel fuel usually only takes up approximately 50 ppm, depending on composition, temperature and emulsifying contaminants present in either the water or the diesel. For conventional diesel, EN 590 limits water to 200 ppm and ASTM D975 limits water to 500 ppm.

After water washing during biodiesel purification (although this is often being replaced by dry ion exchange and absorbent processes), there will typically be more water

present than the limits allow. This can be removed in a number of ways, such as vacuum driers and molecular sieves. During storage and distribution, biodiesel should be kept dry. This is a challenge because many diesel storage tanks accumulate water at the bottom due to condensation. Suspended water is a problem in fuel injection equipment because it contributes to the corrosion of the closely fitting parts in the fuel injection system. Water in biodiesel also enhances microbial growth.

The current ASTM specification uses a centrifuge method and does not reflect dissolved water. The EN specification uses measurement by Karl Fischer (KF) method which measures total water. The ASTM is debating whether to change to the KF method, but there is some debate on its necessity, as a certain amount of water will be picked up during transportation.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Water & sediment, vol%, max	0.05	0.05	None	-	-	-	0.05	-	-	0.05	0.05	-	0.05
Water, ppm, max	-	-	500	500	500	500	-	500	500	-	-	500	-

As shown above, the national specifications all agree that the water content of biodiesel must be below 500 ppm, or water and sediment maximum level combined of 0.05 vol% (500 ppm). The Europeans do not measure water and sediment together as much lower levels of sediment are required with advanced diesel particulate trap technology.

5.1.1.8. Other Contaminants

The alkaline earth, or group 2 metals are soft, low-density metals, such as Calcium and Magnesium and are present in the natural oils. They react readily with halogens found in the environment to form ionic salts. They also react with water to form strongly alkaline hydroxides, although not as aggressively as the alkali metals (Na and K) found in the transesterification catalysts. A limit of 5.0 mg/kg is included for group 2 alkali metals in both the CEN and ASTM specifications to protect exhaust gas catalyst systems. A reduction in this limit is under discussion at CEN.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Metals (Ca + Mg) ppm, max	5	Report	-	5	5	Report	-	5	5	-	5	5	5

Since the cost of test equipment is significant (at approximately US\$40 – 50,000) and most of the APEC economies do not have vehicles with particulate traps at present, the validity of this specification in the short term is debatable. However, these metals may also be linked with fuel instability. Even trace amounts of metal contamination have been found to have a large effect on biodiesel oxidative stability.

Undesired complications in the production process may occur if there are any irregularities in the feedstock. Feedstock at a biodiesel plant should be as consistent as possible. Specification testing should cover all properties whenever feedstock is changed, and similarly, if the process is new or modified. Used oils from the same location can vary substantially. It is often an unknown component in the feedstock, not an elevated level of FFA or phosphorous, which causes problems in the process and/or in the quality of the final biodiesel product.

5.1.2. Addressing Feedstock Effects (Shortcomings)

Studies have sought to overcome any shortcomings in feedstock quality, particularly with regard to tallow and used cooking oils (8). Modifications and additions to the conventional esterification process are required. Biodiesel from tallow requires careful filtration to remove insolubles. Esterification of high FFA tallow required the use of excess methanol and base catalyst.

Generally it has been found that tallow and palm oil derived biodiesel have inadequate cold flow properties. There are three ways of lowering the pour point of palm oil derived biodiesel, namely:

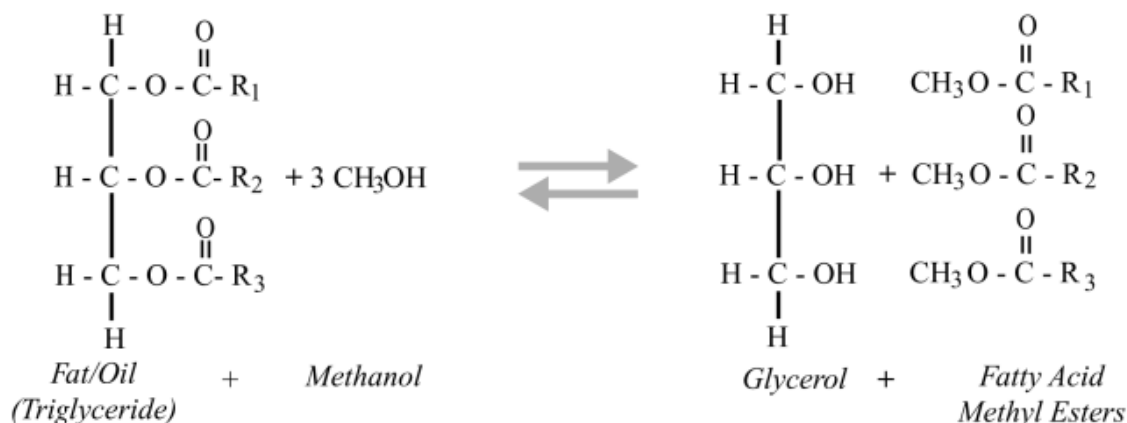
1. use of additives,
2. esterification of palm fatty acids with certain carbon chain length, and
3. fractionation of palm oil methyl esters.

The Malaysian Palm Oil Board (MPOB) has patented technology (Malaysian patent PI 20021157) that allows the production of winter palm diesel with a low pour point of up to -21°C , and even as low as -40°C . However, there is as yet little commercial or market experience of this product and significant yield penalties could be expected.

5.2. Process Dependent Properties

A schematic of the reaction to produce FAME biodiesel is shown in Figure 5.

Figure 5: Schematic of the Transesterification Process to Produce Biodiesel



In the transesterification reaction, the alkoxy group (CH₃O-) of an ester is exchanged with that of a higher alcohol to form a new ester product. In contrast, direct esterification is the reaction between the carboxylic acid group (COOH) of the fatty acid and an alcohol to produce an ester plus water.

A strong mineral base catalyst such as sodium hydroxide (NaOH) or potassium hydroxide (KOH) is needed to initiate and promote the transesterification reaction because the alcohol is only sparingly soluble in the oil phase. After the reaction, the catalyst is neutralized with a strong mineral acid. Direct esterification is normally carried out in the presence of an acidic catalyst such as sulphuric acid (H₂SO₄). Generally, the methoxide base catalyzed transesterification process is preferred because of faster and more complete reaction which takes place at lower temperature and pressure. The acid catalyzed reaction requires temperatures way in excess of the boiling point of methanol (65°C) to be completed in hours, and hence requires a pressure vessel. Furthermore, the acid esterification reaction is in equilibrium, and thus requires higher levels of methanol to drive, and can reverse when the methanol is removed. An approach is to use solid (packed bed) acid reactors, but the use of acid catalysis is still minimal compared to the methoxide (base catalyzed) process.

The critical quality aspects of the process are:

- Complete reaction to consume the triglycerides (also for more favorable economics):
 - Needed to comply with specification on minimum ester content, mono-, di- and triglyceride content, and possibly carbon residue;
- Removal or separation of glycerin co-product:
 - Needed to comply with specification on free and total glycerides and also possibly carbon residue;
- Removal of catalyst (Na and K):
 - These are limited in biodiesel specifications and are particularly important when vehicle O₂ sensors are used and exhaust particulate traps. They may also cause deviation on sulfated ash limit;

- Removal of alcohol (typically methanol):
 - Needed to comply with safety limits on flash point and methanol content which is aggressive to fuel injection system materials;
- Absence of FFA:
 - Needed to comply with acid number. High acid numbers lead to instability and incompatibility with fuel system metals, such as problems with the copper corrosion test;
- Removal of water (drying);
- Cleanliness and filtration:
 - Total Contamination compliance.

5.2.1. Reaction Completion

Complete reaction is reliant on excess methoxide (reacted methanol and base catalyst), methanol and sufficient residence time. To achieve complete reaction to the methyl ester, various reaction schemes are used. They vary from batch to multi-batch (semi-continuous) to continuous and may use varying methanol injection points and times, as well as counter-current flows. A nearly complete reaction is required to comply with the specifications for a number of parameters, such as minimum ester content.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Ester content, wt%, min	96.5	Report	–	96.5	96.5	96.5	96.5	96.5	96.5	–	96.5	96.5	–

A minimum ester content is typically specified to exclude poor quality biodiesel that has been produced using inadequate technology, and containing high amounts of unsaponifiable material and polymers. A limit is sometimes also necessary for Customs where financial incentives are associated with biodiesel.

5.2.2. Washing

The primary purpose of the ester washing step is the removal of any soaps formed during the transesterification reaction. In addition, the water provides a medium for addition of acid to neutralize the remaining catalyst and a means to remove the product salts.

Methanol is toxic, reduces the flash point, and is aggressive to fuel system components, mainly elastomers, hence the residual methanol should be removed before the wash step. This prevents the addition of methanol to the wastewater effluent. However, some processes remove the methanol with the wash water and then flash separate it from the wash water.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Methanol, wt%, max	0.2	0.5	–	0.2	0.2	0.2	–	0.2	0.2	–	0.2	0.2	0.2 vol%

The ASTM standard sets the flash point specification at 93°C minimum in order for biodiesel to be classified as a non-hazardous material for shipping in the U.S. The methanol specification by the GC method is set at 0.2 vol% maximum. However, if the flash point of the biodiesel exceeds 130°C, the ASTM standard does not require the measurement of methanol content.

Softened water, which is slightly acidic, eliminates calcium and magnesium contamination and neutralizes remaining base catalysts. Similarly, removal of iron and copper ions eliminates a source of catalysts that decrease fuel stability.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Ash, wt%, max	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.01	0.02	0.02

A maximum ash limit of 0.02 wt% is a general approach in national specifications. However, a biodiesel fuel containing this maximum level of ash would pose serious problems for the durability of diesel particulate filters. The diesel fuel in the real world contains ash at several magnitudes lower than this level, so vehicle manufacturers are asking to reduce the ash content in automotive fuels by a factor of 100.

5.2.3. Other Ester Treatments

Absorbents, such as magnesium silicates and silica gels are used to selectively absorb hydrophylic (highly polar) materials such as glycerol and mono- and diglycerides.

The comparison table below shows that the most common standards approach is to limit the free glycerol and total glycerol, rather than examining the breakdown of glycerides into mono-, di- and triglycerides. However, the CEN believes individual limits are necessary to describe the quality of biodiesel for operability over a wide range of conditions. Triglyceride content is a good indicator of pure uncovered oil, whereas monoglycerides are suspected in low temperature storage tank deposits. In fact a lower monoglyceride content specification may be considered by CEN. The free glycerol (also commonly called glycerine) content is actually calculated from the mono-, di- and triglyceride specification and is arguably redundant.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Monoglycerides, wt%,max	–	Report	–	0.8	0.8	–	–	0.8	0.8	–	–	0.8	–
Diglycerides, wt%,max	–	Report	–	0.2	0.2	–	–	0.2	–	–	–	0.2	–
Triglycerides, wt%,max	–	Report	–	0.2	0.2	–	–	0.2	–	–	–	0.2	–
Free glycerol/glycerin, wt%,max	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02	–	0.02	0.02
Total glycerol/glycerin, wt%,max	0.25	0.38	0.24	0.25	0.25	0.25	0.24	0.25	0.24	0.24	0.24	0.25	0.24

The above chemical composition limits should ideally all be replaced by performance based limits such as oxidation and thermal stability in the APEC biodiesel standard. However in the absence of sufficient data to develop suitable test methods and set appropriate limits, the OEMs and FIEs in those markets with sophisticated vehicle emissions control systems require chemical composition limits.

Some vegetable oils and many yellow greases and brown greases leave an objectionable color in the biodiesel. There is no color requirement in biodiesel specifications, but activated carbon treatment can lighten the color of oils and biodiesel.

5.2.4. Glycerol Fraction Separation, Properties and Processing

As can be seen from the reaction, a major byproduct of biodiesel production is glycerol (also commonly referred to as glycerin), a 3-carbon carbohydrate. Separation of the biodiesel from the raw glycerol byproduct is generally initially accomplished by gravity. The amount of raw glycerol is approximately 16 wt% to 18 wt% of the input oil. The contaminants of the raw glycerol byproduct are those used in the transesterification reaction, namely methanol and base (NaOH or KOH), salts, water, unreacted methanol and triglyceride, as well as reaction products, including FAME and alkaline soaps. The composition of the raw glycerol is not stable and depends on several factors including the acidity of the input oil. It is a dark brown-green substance. The further processing and purity of the glycerol is determined by the market for this co-product. This is not addressed further here, save to note that it is an important consideration, economically and quality-wise, for biodiesel producers and their derivative customers.

5.3. Biodiesel as a Performance Fuel

A number of parameters that are important to the final biodiesel and mineral diesel fuel blend are specified in some standards for the biodiesel itself. This can exclude biodiesel produced from certain feedstocks. Since the APEC biodiesel standard is to be

developed for biodiesel as a blend component, typically up to B5, caution should be exercised when defining diesel fuel performance limits for biodiesel.

5.3.1. Antifoaming

Pure biodiesel has excellent anti-foam properties, better than petroleum diesel (11). This enables and ensures fast filling of vehicles, without possible foam leaks or overflows.

5.3.2. Cetane Number

Cetane number (CN) is one of the most common indicators of diesel fuel quality. It measures the readiness of the fuel to auto-ignite when injected into the engine. It is generally dependent on the composition of the fuel and can impact the engine's startability, noise level and exhaust emissions, with a high CN being desirable. The CN of biodiesel is generally observed to be quite high. Data shows values varying between 45 and 70, as compared to 40 to 52 for typical mineral diesel fuels. The CN of biodiesel depends on the distribution of fatty acids in the original oil or fat from which it is produced. The longer the fatty acid carbon chains and the more saturated the molecules, the higher the CN.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
CN, min	51	Report	49	51	51	51	51	51	51	42	–	51	47

Many standards set the CN of biodiesel at the same limit as for finished diesel fuel. However, this practice could exclude biodiesel produced from soya or jatropha oils for example, which typically have a CN below 50. As a blending component with mineral diesel oil the final Bx product could meet or exceed the cetane number requirements applicable in the market. Hence a lower CN for biodiesel which is to be used as a blending component is desirable.

5.3.3. Chemical Structure and Oxygen Content

Biodiesel molecules typically consist of about 19 carbon molecules, the C18 fatty acid reacted with methanol containing 1 carbon molecule and 2 oxygen molecules of the ester. This means that the oxygen content is typically of the order of 11%.

$$\text{Oxygen Content} = \frac{\text{Oxygen} : 2 \times 16}{\text{Oxygen} : 2 \times 16 + \text{Carbon} : 19 \times 12 + \text{Hydrogen} : 19 \times 2}$$

Diesel does not contain oxygen and typically has 12 to 22 carbon atoms per molecule. A further difference is that biodiesels are predominantly straight chain hydrocarbons esters, whereas diesel contains ring structures, such as aromatic molecules. Straight chain molecules have higher cetane numbers and hence, easier and quicker ignition, which contributes to smoother combustion in diesel engines. This is because the

combustion in diesel engines takes place by chain breaking leading to free radicals that combust with oxygen. Straight chains break more easily than ring structures.

5.3.4. Cold Flow Properties

For mineral diesels, each component has its own crystallization temperature, so solidification is a gradual process, whereas B100 biodiesel tends to be a much simpler mixture containing relatively few components, so that one or two components tend to dominate, such that solidification is much more rapid and much more difficult to control. The temperature at which B100 starts to gel varies significantly depending on the mix of esters and therefore the feedstock oil used to produce the biodiesel. For instance, low erucic acid RME starts to gel at -10°C and biodiesel from tallow gels at approximately +16°C.

Given that the ultimate criterion for cold flow is fit for common purpose, the cold filter plugging point (CFPP) limit is arguably the most appropriate indicator, as it shows when a filter would start plugging or blocking. The CFPP of biodiesel or blends of biodiesel with diesel should align with that of conventional diesel in a particular distribution region, as per the winter or summer periods. This is based on analysis of the ambient temperatures in those regions. This can be achieved by blending and/or use of cold flow improver additives (9). There is some concern that this test, developed for petroleum diesel, may not be so suited for biodiesel. However, this is not observed from international experience. In Europe, where biodiesel is most widely used and has many areas with low ambient temperatures, conventional CFPP testing and limits continue to be successfully used for biodiesel.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
CFPP, °C, max	-	-	Report	0	+5 to -44	-	-	-	-	-	0	-	-
Cloud Point, °C	-	-	-	-	-	-	-	-	-	Report	-	-	Report

A note on diesel specifications is that often the CFPP must be no more than 10°C below the cloud point. This is not as important for biodiesel because typically being a more pure chemical, the CFPP is closer to the cloud point. The difference is roughly the same for palm oil methyl ester, and 5°C lower for rapeseed and soy oil methyl esters.⁽¹⁵⁾

5.3.5. Conductivity

Pure biodiesel, due to its polarity has excellent conductivity, greater than 500 picoS/m, and therefore reduces the risk of static induced sparks and fires (11).

¹⁵ Source: Afton Chemicals.

5.3.6. Corrosion

The water absorption coupled with the presence of oxygen tends to contribute to increased corrosion, but this is countered by the wettability that reduces oxygen transfer to metal surfaces. The copper corrosion test focuses on sulfur compounds that are aggressive to copper and yellow metals. Sulfur, and specifically the corrosive forms thereof, are absent in biodiesel, and compliance to diesel specification limits is typically easily achieved⁽¹⁶⁾.

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Cu Corrosion 3hr@50°C, Class X, max	1	-	1	1 (at 100°C)	1	1	3	1	1	3	1	1	3

5.3.7. Density

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Density, kg/m ³ , min-max	860–890	Report	820–900	860–900	860–900	860–900	850–890	860–900	860–900	-	860–900	860–900	-

Biodiesel tends to have higher density, in the range of 860 to 900 kg/m³, compared to a typical range of 800 kg/m³–860 kg/m³ for mineral diesels. The higher side is associated with higher molecular mass of the longer chain alkyl ester molecules. The density limits, as required in many economies may therefore limit very light or heavy molecules, and hence feedstock oils. However, such non-compliant oils do not commonly occur, so the limit of 860 kg/m³–900 kg/m³ seems appropriate until there is a challenge or proposal to broaden this. The lower limit should be easier to reduce, if required, as it is typically 800 kg/m³ or 820 kg/m³ for conventional petroleum diesel.

Table 5: Typical Properties of Biodiesel Compared to Diesel

	Density g / cm ³	Caloric value MJ / kg	Caloric value MJ / dm ³
Biodiesel	0,88	38,4	33,8
Diesel	0,83	42,9	35,6
Variation		-11%	-5%

Source: Terry de Winne, "Biofuels for Sustainable Transport," <http://www.biofuels.fsnet.co.uk> and National Biodiesel Board

The upper density limit for petroleum diesel also controls the heavier aromatics and high molecular weight paraffins that tend to increase particulate matter (PM) emissions. As biodiesel does not contain these heavy molecules it is less sensitive to maximum boiling

¹⁶ Personal communication: Dr. Irene Finnegan, Bioservices Laboratories.

range and a maximum density specification. Nevertheless, the EN 14214 specification has maximum limits of 900 kg/m³ and the U.S. ASTM has a maximum T90 of 360°C. Both these limits may be raised for biodiesel without a significant effect on PM emissions, relative to conventional petroleum diesels. However, as biodiesel molecules tend to be similar and high boiling, these high boiling materials can cause engine oil dilution and contamination. This leads to poorer lubrication and ultimately higher engine wear rates and possible catastrophic failures. The need for raising these limits or providing for a waiver would probably depend more on whether the current accepted limits cause undue hardship to biodiesel producers in APEC economies.

5.3.8. Viscosity

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India	Indonesia	Japan	New Zealand	The Philippines	Korea	Thailand	U.S.
Viscosity, cSt, min-max	3.5–5.0	Report	1.9–6.0	3.5–5.0	3.5–5.0	2.5–6.0	2.3–6.0	3.5–5.0	2.0–6.0	2.0–4.5	1.9–5.0	3.5–5.0	1.9–6.0

The minimum viscosity limit is typically driven by considerations of lubricity and leakage from injectors. On the other hand, high viscosity diesel can cause poor injector spray atomization which can lead to excessive coking and oil dilution. Typical ranges are 2 mm²/s – 5 mm²/s at 40°C for conventional diesel. Given that biodiesel has excellent lubricity, there is no reason for it to have a different lower limit than conventional diesel. The European minimum limit of 3.5, as opposed to 2.0 for normal diesel thus appears too stringent and excludes CME. The ASTM minimum limit of 1.9 mm²/s at 40°C, or the Brazilian approach that focuses on the final blend being acceptable according to the standard diesel specifications both appear more reasonable. In practice, the viscosity of some biodiesels tend to be higher (fatty acid feedstock dependent). The maximum viscosity limit of the ASTM D6751 standard is 6.0 mm²/s at 40°C versus 5.0 mm²/s per EN 14214. This maximum limit needs to be guided by injector spray patterns; given that biodiesel contains less heavy, high boiling point material than conventional diesels, and the approximate 11% oxygen, there is reason to consider allowing a higher maximum viscosity limit than for conventional diesel, with the final blend complying to the typical globally accepted maximum limit for conventional diesel of 4.5 mm²/s at 40°C.

Higher viscosities result in larger droplets being injected and contribute to less complete combustion and hence fuel dilution of the lubrication oil. Engine oil viscosities are typically of the order of 100 mm²/s at 40 °C versus approximately 10 mm²/s for uncombusted diesel, and as viscosity blending is dominated by the lighter component, a small percentage of biodiesel dilution results in significant loss of viscosity, and hence susceptibility to bearing damage. Bearing loadings are typically high in diesel engines due to the high torque, and failures may result. However, U.S. experience has shown that the higher limit of 6 mm²/s for biodiesel, particularly when used in B20 blends, has not resulted in poor spray patterns. This may not be the case for passenger cars (light duty diesels) as the smaller bore makes these generally more susceptible to fuel

dilution.

Note should be taken that the CEN standard EN 14214 requires that viscosity of biodiesel should not exceed $4.8 \text{ mm}^2/\text{s}$ below -20°C , when the CFPP is less than -20°C . This precaution is required to protect the injection pump drive from damage due to excessively high fuel viscosity generating loads in the pump drive train.

5.4. Blends with Diesel & Additization

Waivers from the diesel specifications for biodiesel blends are not common internationally. This is because the focus is on making good quality fuels. Other than cold properties where it will be important to adjust the base diesel quality to ensure "fit for purpose" blended product, it should not be difficult to remain on specification when blending biodiesel as long as the biodiesel complies to acceptable quality standards. Additization provides a cost effective solution in many cases. Tests of the final blend quality against the specification must however be carried out as part of the quality monitoring process.

Initially, when biodiesel volumes were low, the additives that were established for mineral diesels were tested and used where they showed benefits. Often these needed higher treat rates, as they were not designed and matched to biodiesel chemistry. As biodiesel usage has increased, specialized or selected and optimized additives have been developed to provide better performance and/or lower treat rates. Most work has been focused on rapeseed methyl ester, as this is the highest volume biodiesel globally⁽¹⁷⁾ and Europe has the most stringent diesel performance needs.

The main additive companies supplying petroleum diesel additives and vegetable oil additives are Lanxess, Ciba, Eastman, Lubrizol, Degussa, Afton Chemical Corporation, BASF and Innospec. These companies have over recent years developed and proven additives for biodiesel and biodiesel blends, and they have generated test data that they willingly share (11), particularly to customers and potential customers. The customers, or biodiesel producers and marketers need to test their biodiesel and the diesel blends with the recommended additives to confirm the resultant property improvements. In addition, particularly where higher treat rates are used than applies to conventional diesel, "no harm" testing is required. This should be backed up by monitoring in the market.

A number of issues still need to be addressed, such as:

- ensuring that biodiesel and biodiesel blends can comply with market demands with availability of suitable additives;
- the additives need to be able to cater for biodiesel variations, caused by different feedstocks, production methods and different blends;
- test methods for petroleum based fuels that correlate with performance, need to also apply to biodiesel.

¹⁷ Europe uses approximately 70% of global biodiesel and most of this is based on RME.

Two of the major obstacles to widespread acceptance of higher percentage biodiesel blends being used are;

- stability, and
- low temperature operability.

These can be addressed by a combination of feedstock selection, proper processing and quality control, as well as by the use of appropriate fuel additives.

5.4.1. Antifoaming

The polar nature of biodiesel reduces surface tension and breaks (actually destabilizes) foam bubbles. B100 and blends show excellent short foam decay times, similar to base petroleum diesels with anti-foam additive included. Although B100 has low foaming tendencies, blends tend to have increased levels of foaming resulting from negative synergy with diesel. This is, however, blend dependent. Therefore while antifoam additives are typically not required in biodiesel blends, if problems are experienced, these are normally easily corrected with such additives (11).

5.4.2. Cetane

Although most B100 products have high natural CN, while blending with diesel, the change is non linear. The blends do however respond normally and consistently to conventional cetane improver additives according to additive suppliers, such as Afton Chemical Corporation and Innospec. (11)

5.4.3. Cold Flow Properties

The cold flow properties, cloud point and CFPP, of varying biodiesel blends are non-linear compared to their blend percentages. The given blend level and biodiesel and mineral diesel need to be tested, such that a suitable CFPP additive can be added at the necessary dosage rate. For low-level blends, most biodiesel types, even those with high CFPP's should be able to be treated with additives to achieve a marketable result (11). Cold flow improver additives typically work by preventing nucleation of heavy molecules. However, while cold flow improver additives have some positive effects, they cannot overcome problem biodiesels, such as palm oil methyl ester with CFPP of 10°C, which typically significantly exceeds market requirements. Additive companies such as Clariant, Infineum, Innospec, Afton Chemical Corporation and Lubrizol have products and continue with tests of new products, and in different biodiesel basestocks, but many biodiesel producers report limited success.

5.4.4. Conductivity

Fuel conductivity is a safety issue, to prevent electrostatic ignition (11). As stated previously in this report, biodiesels have excellent conductivity, such as soy-derived B100 being of the order of 500 pS versus typical hydrotreated low sulfur (50 ppm) diesel having values below 100 pS, and as low as 5 pS. Adding B5, B10 and increasing amounts of biodiesel increases conductivity roughly linearly. B100 from palm oil has a value of 100 pS, which is similar to mineral diesel. Conventional conductivity

additives are found to work well with biodiesel and biodiesel blends. Blends higher than B20 should be tested to confirm conductivity. B20 and lower should be treated with fuel conductivity improver additives.

5.4.5. Density

Density for blends of biodiesel with petroleum diesel roughly follow a linear equation, and it is thus possible to determine the resultant density of a diesel/biodiesel blend if the density of the two components and the blend concentration is known (18). For low-level blends, such as B5, the impact of biodiesel within tolerances will not cause the resultant blend to be off-specification. Biodiesel can be useful to blend with low-density diesel components, such as FT diesel, to achieve the required finished diesel fuel density. A caution though is that the resultant blend will have a lower energy content leading to higher fuel consumption, although this is typically acceptable as most diesel does not have an energy content specification.

5.4.6. Lubricity

At low-levels, biodiesel being a polar ester, is an effective replacement for diesel lubricity additives that need to be added to low sulfur diesels. Tests by various parties have shown that at 1%–2% (B1–B2) the high frequency reciprocating rig (HFRR) wear scar diameter drops to around 300 micrometers, versus a most strict limit for conventional diesel of 400 micrometers. This is a benefit, and it represents value addition to the blend. Initially economies that do not have capacity to produce biodiesel may therefore benefit by importing low-levels of biodiesel (typically 1%–2%) to replace diesel lubricity additives. The benefit is of the order of US\$0.1 per liter, as a premium to the diesel price.⁽¹⁸⁾ The ASTM D6751 standard allows biodiesel blend components to contribute to improved lubricity.

5.4.7. Viscosity

Viscosity does not have linear blending properties. However, the non-linear effect is insignificant for low-level biodiesel blends, up to B5 (18). For higher-level blends, actual blend viscosities need to be tested or calculated using proven models.

5.4.8. Stability (Oxidation)

The process of oxidation of all organic molecules, including conventional diesel and biodiesel, typically follows a chemical reaction as follows:

- unsaturated bond loses hydrogen → free radical,
- free radical → fatty acid peroxide radical,
- fatty acid peroxide → hydroperoxide, and
- hydroperoxide → aldehydes, ketones and polymers.

¹⁸ Christo Cloete, Fuels technical manager, PetroSA, South Africa.

This process can be halted by an antioxidant converting the original free radical back to the fatty acid by donating hydrogen. Hindered phenols and aromatic amines are commonly used as anti-oxidant additives. These also contain metal deactivators, as contact with metals catalyses biodiesel degradation.

Test work by the ASTM oxidation work group has shown that despite biodiesel complying with EN 14214 Rancimat induction period of 6 hours (volatile acids build up time), the mixture of B5 often fails the ASTM D2274 oxidation test, measuring insolubles after aging. Therefore blending has negative synergy. It was also found that there is not a good correlation between the European “Rancimat” and U.S. deposits test. The U.S. biodiesel complies with their deposits limit, but typically meets only 2 hours, versus a requirement of 6 hours in the Rancimat test. In Europe the biodiesel industry has now moved to anti-oxidant addition at manufacture. Additive companies, such as Afton Chemical, Ciba, Degussa and Innospec have presented data showing passes for B5, B10 and B20 with additives.

The ASTM working group has identified major factors influencing stability as follows:

- presence of partially reacted or un-reacted oils,
- level and type of un-saturation, 18:2 and 18:3 in particular for soy oil methyl esters as tested, and
- presence of anti-oxidants, either natural or added.

They also found that most market problems for B20 are due to off-specification biodiesel and are not a general stability requirement problem.

Biodiesels and blends in particular need tailor-made additives or mixtures thereof, depending on the feedstock used to produce the biodiesel. Some additives that work well with European rapeseed biodiesel do not perform well with soy biodiesel. It has also been observed that some additives have performed differently among the same kinds of biodiesel produced from the same feedstocks but in different regions (19) and using different processes (7). Development work continues on oxidation tests and additives. Companies that offer such additives include Lanxess, Ciba, Eastman, Lubrizol, Degussa, Afton Chemical Corporation and Innospec.

5.4.9. Performance Additives

Performance additives are typically not related to specification requirements, but rather utilized for branded products to support performance or marketing claims. Some examples follow.

5.4.9.1. Corrosion Protection (Inhibitors)

These additives are added to protect logistics systems and particularly fuel systems. They have the added benefit of protecting the biodiesel from degradation and acid formation since those reactions are catalyzed by metals.

5.4.9.2. Injector Deposits (Detergents)

The most common test for diesel fuel is the Peugeot XUD-9 injector flow reduction test. It has a limit of 15% minimum, and failures with base diesel and B5 based on soy methyl ester have been reported. When the blend was increased to B20, and B100, pass values were achieved. This is due to the solvency properties of biodiesel, acting as a natural detergent. According to Afton Chemical Corporation and Innospec, conventional detergent additives have been found to be effective in a range of biodiesel types and blends, giving more than 50% improvement.

5.4.9.3. Water Separation (Demulsifiers)

The polar nature of biodiesel means that it tends to absorb water and this can accelerate degradation, growth of bugs (microbes), and corrosion. Being hygroscopic, biodiesel fuels require special handling to prevent high water ingress. Emulsification is typically worsened by presence of contaminants, including oxidation products. Demulsifier additives are very seldom used, as it is better to treat the root cause as outlined in section 5.1.1.7 above, rather than to try to treat the symptom.

6. Vehicles Compatibility and Emissions

Historically it was intended to produce biodiesel in a kind of closed-loop from agricultural products for the agricultural machinery, mainly tractors, and the farming sector pushed for its use with equipment suppliers. Hence, the first warranties issued were for tractors or Combines (e.g. Same, Steyr, John Deere, Massey-Ferguson, Lindner etc.). With the development of more sophisticated marketing strategies, the focus has extended to other diesel-driven vehicles such as buses in the public transport fleets of cities, taxi fleets, and more sensitive users, including the leisure marine sector, underground mines, and certain private car owners. A further extension was achieved by obtaining warranties for the new generation of modern, high-pressure fuel injection systems such as the common-rail systems (e.g. Mercedes-Benz, Peugeot, and Volkswagen (VW)) (20).

In 1996, the CEO of Volkswagen AG, Dr. Ferdinand Piëch, declared full support to biodiesel by assuring the provisions of warranties for nearly all diesel models of the group including the brands AUDI, SEAT, SKODA and VW (21). Since the VW approval, use grew significantly, and today more than 400,000 cars use pure biodiesel (B100) in Germany, Austria and Sweden. In contrast, 10-15 million cars use biodiesel as an extender (B2) to fossil diesel in France. A “middle of the road” approach is the strategy adopted by Chechnya and Slovakia, and also in France, which offer fossil diesel blended with 30 vol%–40 vol% biodiesel.

The U.S. OEM’s views can be summarized as follows:

- B100 Must Meet ASTM D 6751;
- Most OEMs have B20 experience, so they won’t void warranties, but problems caused by the fuel are the responsibility of the fuel supplier;
- They want to see additional experience in the field; and
- Higher blends are accepted based on experience of OEMs and their technology

The World Wide Fuel Charter (WWFC) is prepared and supported by the Alliance of Automobile Manufacturers (AAM), ACEA, Engine Manufacturers Association (EMA) and Japanese Automobile Manufacturers Association (JAMA). The WWFC is a statement by the world’s auto industry on the quality of fuels needed to ensure optimal operation of different types of vehicles they manufacture. Recommended specifications are provided for four different categories of gasoline and diesel fuels.

The WWFC (2006) allows the addition of biodiesel up to 5 vol% in Fuel Categories 1-3, and thus guarantees vehicles using biodiesel at these blends with the following caveat; for the biodiesel, both EN 14214 and ASTM D6751, or equivalent standards, should be met. Where biodiesel is used it is recommended that fueling pumps be marked accordingly. For Category 4 fuels, the WWFC calls for biodiesel to be non-detectable (i.e. at or below detection limit of the test method used, which is specified as EN 14078). This means that automakers prefer not to have biodiesel in the most advanced vehicles and thus some may reserve the right not to repair a vehicle under warranty if the origin

of the problem was perceived to be poor or inappropriate fuel quality resulting from the use of biodiesel.

Generally, biodiesel is believed to enhance the lubricity of conventional diesel fuel and reduce PM emissions. At the same time, engine, auto and fuel injector manufacturers have concerns about introducing biodiesel into the marketplace, especially at higher levels, because biodiesel may be less stable than conventional diesel fuel. Their view is that precautions are needed to avoid problems linked to the presence of oxidation products in the fuel. Some fuel injection equipment data suggests such stability problems may be exacerbated when biodiesel is blended with ultra-low sulfur diesel fuels, as it is known that the natural antioxidants, or free radical scavengers, are often removed by the desulphurization process, leading to lower stability. This can generally be overcome by the addition of appropriate antioxidants.

The transition from diesel to biodiesel blends that have higher solvency, may lead to an especially large increase in sediments that could plug fuel filters.

Fuel system parts must be specially chosen for their compatibility with biodiesel. More modern diesel fuel systems are typically designed and specified to be compatible with biodiesel at low-levels such as B5.

6.1. Diesel Vehicle Requirements

Biodiesel in its neat form is likely to have materials compatibility issues with current on-road vehicles. These issues are discussed below with reference to fuel system effects, engine performance and emissions. The extent of these problems is related to the level of biodiesel blends and applies to experience with biodiesel that meets either the U.S. ASTM or European biodiesel specifications. The diesel fuel used for blending complies with the specifications for those regions.

6.1.1. Fuel System Effects

The common position statement issued by the diesel fuel injection equipment (FIE) manufacturers for their equipment compatibility with biodiesel is summarized in Table 6.

Table 6: Biodiesel Blends Compatible with FIE Manufacturers and their Quality Requirements

	B1 – B5	B6 – B100	Specification
Bosch	B5, EN		EN 14214
Siemens	B5, ASTM, EN		EN 14214
Delphi	B5, ASTM, EN		EN 14214
Stanadyne	B5, ASTM, EN	B20, ASTM	EN 14214
Denso	B5, EN		EN 14214

6.1.1.1. Elastomers

Hoses and seals (termed elastomers) tend to absorb some of the petroleum diesel, and this typically leads to swelling. The oxygen- and hydroxyl (-OH) containing methyl esters have strong solvency properties with respect to natural rubber and several soft plastics. As a result, old rubber fuel lines and some seals or gaskets on fuel tanks may slowly

deteriorate in the presence of higher concentrations of biodiesel. Few of these solvency effects are noticed up to a B20 blend, and most problems such as shrinking occur when using B100. This is elastomer dependent, but polar biodiesel typically dissolves more in polar type elastomers, such as nitrile butadiene rubber seals in fuel systems (tank, hoses, seals, fuel pumps, injectors).

This is not a major problem with post-1994 vehicles using low-level biodiesel blends. With older vehicles, flexible fuel lines and return lines tend to slowly swell when in contact with biodiesel. General purpose nitrile rubber, high aceto-nitrile rubber, peroxide-cured nitrile rubber, fluorocarbon filled with carbon black and fluorocarbon without carbon black elastomers are compatible with B20 and do not exhibit significantly different break load, dimensions or volume than when exposed to diesel (25). The switch to low sulfur diesel has caused most OEMs to switch to components suitable for use with biodiesel, but users need to get specific information from OEMs. Manufacturers recommend that natural or butyl rubbers not be allowed to come in contact with pure biodiesel (26,27,28,29). For RME and SME based B20, fluorocarbon elastomers of medium to high fluorine content were found to be the most compatible (30).

6.1.1.2. Filters

The average molecular weight of biodiesel is approximately 30% higher than that of No. 2 diesel fuel. The larger molecules tend to precipitate out at higher temperatures than those of diesel fuel, leading to problems of filter plugging in cold weather (4). Conventional diesel leaves deposits in the tank and fuel system. Biodiesel cleans up the residues that the diesel leaves behind. If a vehicle has been left standing for a long time with diesel fuel in the tank, the bottom of the tank may have rusted (free water separation, mainly due to temperature dropping, is a common problem with diesel fuel). Biodiesel absorbs water, drying and loosening rust, and this could clog or shorten the life of fuel filter (4,7,19,22), particularly during the transition phase. This problem is addressed by increasing the filter change frequency in vehicles using biodiesel blends (23), particularly during transition. B5 and B20 do not cause filter blockage problems when the blended biodiesel is oxidized and contains low water and sediment content (30). There is only a 2% chance of filter blockage problem when using B5, requiring an additional filter change when first switching to B5 (24). Consumers tend to have enough warning in the form of reduced engine performance before critical filter clogging in older vehicle technologies. However, in modern common rail engines, a drop in rail pressure due to filter clogging can lead to a sudden engine stop, as the minimum protective pressure for lubricity is lost.

6.1.1.3. Injector Systems

New diesel engines are common rail with unit injector systems characterized by electronic control with small solenoid valves and sensors, and by much higher pressures. The older engine systems using distribution pumps have been tested with good results on biodiesel. The higher-pressure systems are less generic than older systems. Therefore, it is difficult to extrapolate results for one manufacturer to cover a range of engines (20). However, fuel injection equipment manufacturers have issued

a joint statement approving the use of B5, where the biodiesel component meets the EN 14214 specification.

6.1.1.4. Metals

Some metals, such as brass, bronze, copper, lead, tin and zinc may have a catalytic effect on the biodiesel oxidation process. Contact with these materials should be avoided, particularly in long-term storage (10). The presence of Na, K, Ca and Mg can also increase injector deposits.

6.1.1.5. Paint

As explained above, biodiesel is a good solvent. If left on a painted surface long enough, it can soften certain types of paints (paints are typically polar, and like dissolves like). Therefore, it is recommended that any biodiesel or biodiesel blend spilt onto painted surfaces should be wiped or washed off immediately (4,26).

6.1.2. Engine Performance

Table 7 and Table 8 outline the level of support from various vehicle manufacturers towards the use of biodiesel.

Table 7: Biodiesel Blends Approved for Use by Manufacturers and their Quality Requirements

	B1 – B5	B6 – B100
BMW	Not Recommended	
Case IH	B5, All vehicles	B20 and B100, select vehicles
Caterpillar	B5, Most engines, ASTM, EN	B20, B100 in select models
Cummins	B5, All engines, ASTM	B20, Post-2001, ASTM
DaimlerChrysler	B5, All vehicles, ASTM	B20, Some Post-2006, ASTM
Detroit Diesel	B5, All engines, ASTM and DDC fuel specification	
EMA	B5, ASTM	
Ford	B5, ASTM and EN	
General Motors	B5, ASTM	B20, available as a special equipment option
Holden	B5	
Holden Rodeo	Not recommended	
International		B20, ASTM
Isuzu Commercial Truck	B5	
Jaguar	Not Recommended	
John Deere	B5, ASTM or EN	B20, engines through Tier 3/ Stage III A models
Kubota	B5, ASTM	
Kia	Not Recommended	
Land Rover	Not Recommended	
Mack Trucks	B5	

Mazda	B5	
Mercedes-Benz	B5, All CRDI engines, ASTM (with Oxidation Stability = 6 hr), EN	
Mitsubishi	At Owners Risk	At Owners Risk
New Holland	B5, ASTM	
Nissan Diesel	B5, All trucks, ASTM	
PSA Peugeot Citroën	B5, EN	B30, Most models
Scania	B5 RME	B100 RME in new engines with unit injectors
Toro	B20, 2008 and later models	
VW	B5, ASTM	
Volvo	B5, ASTM	
Volvo Trucks	B5, All engines, ASTM or EN	

Note: Individual regions and model compatibility might vary.

Source: Individual Manufacturers, McCormick, R. (39), National Biodiesel Board, Engine Manufacturers Association (40)

Table 8: Brand Type Models with Warrantees for B100

Audi personal cars all TDI-models since 1996
BMW personal cars model 525 tds / 1997 and 3er + 5er since 2001
Case - IH tractors all models since 1971
Claas combines, tractors warranties exist
Faryman Diesel engines warranties exist
Fiat-Agri tractors for new models
Ford AG tractors for new models
Holder tractors warranties exist
Iseki tractors series 3000 and 5000
John Deere tractors warranties since 1987
John Deere combines warranties since 1987
KHD tractors warranties exist
Kubota tractors series OC, Super Mini, O5, O3,
Lamborghini tractors series 1000
Mercedes-Benz personal cars series C and E 220, C 200 and 220 CDI, a.o.
Nissan lorry, bus, personal car, series BR 300, 400,
Unimog since 1988, a.o.
Primera since 2001
Same tractors since 1990
Seat personal cars all TDI-series since 1996
Skoda personal cars all TDI-series since 1996
Steyr tractors since 1988
Steyr boats series M 16 TCAM and M 14 TCAM
Valmet tractors since 1991
Volkswagen personal cars all TDI- series since 1996
Volkswagen personal cars all new SDI-series (EURO-3)
Volvo personal cars series S80-D, S70-TDI and V70-TDI

Source: KÖRBITZ, W; New Trends in Developing Biodiesel, presentation at Asia Bio-Fuels, Singapore, April 22-23 2002

6.1.2.1. Cold Flow

Biodiesel gels at a higher temperature than diesel, and in certain cases has resulted in starting problems below zero, more noticeably below -5°C (20). Low-level blends are less affected as the broad range diesel components tend to reduce congealing or solidification. Also cold flow improver additives in diesel are often effective with biodiesel blends. This is more of an issue with B100. Impact of different feedstocks is different, with animal source feedstocks (lard, tallow) resulting in higher biodiesel viscosities than vegetable sources (soy, canola) (8,24). This issue can be addressed by using engine block or fuel filter heaters (23,28). Pour point depressant additives have also been found to be effective with biodiesel blends although their effectiveness with pure biodiesel has been reported to be minimal (4). Another suggested method is to add a lighter petroleum fraction such as kerosene (19).

6.1.2.2. Cylinder heads

While deposits with the use of biodiesel are found to be easier to clean than diesel deposits, this varied depending on biodiesel feedstock. The level and type of unsaturates probably affect the nature of the deposit formed. For instance, jatropha-derived biodiesel forms lower amounts of cylinder head deposits than palm oil derived biodiesel (35,36).

6.1.2.3. Engine Oil

Biodiesel, typically being of higher viscosity and having a higher average molecular mass, can often burn incompletely, leading to increased dilution of the engine (crankcase) oil. While biodiesel is approximately 50% more viscous than diesel resulting in larger spray droplets in the combustion chamber, these droplets move past the piston rings to reach the crankcase, thus contaminating the oil (4). Thereafter wear increases, and the greater level of wear metals in the oil leads to accelerated lube oil breakdown (33). Due to its higher average molecular mass, and hence lower evaporative rate, it can accumulate in engine oil during low-load operations (34). This problem is addressed by more frequent oil change intervals. The change is not generally required while using blends up to B20 (24) in the U.S. In Europe, oil dilution is a significant problem with cars equipped with certain types of diesel particulate filter (DPF) systems, and the current maximum allowable biodiesel in EN 590 is 5 vol%. The problem is exacerbated by late injections for cooling and DPF regeneration. This problem will have to be solved before many European OEMs agree to more than 5 vol% biodiesel in EN 590.

6.1.2.4. Fuel Economy

Biodiesel has approximately 5-7% lower energy content than diesel. This reduces the fuel economy of an engine operating on biodiesel or its blends with diesel. The peak power output of the engine is reduced proportionally to the reduction of fuel volumetric heating value, becoming an issue on those occasions when maximum power is needed

from the engine. This is partly countered by the typically greater volumes of product gases from biodiesel combustion.

A B20 blend has been found to reduce fuel economy by approx 12% (4,19,23,31). This appears higher than the 5% that can be explained theoretically. Further, this reduction in fuel economy may not be significant as energy content of diesel fuel can vary from supplier to supplier and summer to winter by as much as 15% (24), and is not controlled by specification limits.

6.1.2.5. Lubricity

Removal of diesel sulfur reduces lubricity, thus requiring additives. Due to biodiesel's polarity and hence lubricity, amounts as small as 1 – 2 vol% can restore the lubricity of U.S. ULSD (4,8) to the specification requirements, and attain an HFRR wear scar of 400 micrometers maximum. 1 vol% of biodiesel can improve the lubricity of diesel fuel by approximately 65% (32).

6.1.2.6. Maintenance Cost

There is a statistically insignificant change in the maintenance cost of vehicles using B20 over the useful life of the vehicle (31,36).

Most European automotives with model years post-1996 are compatible with low-level blends of biodiesel (37,38). Economies such as France are selling B5, and the Czech Republic B30. Nissan Austria has approved its Primera for use with 100% biodiesel. A few medium-aged trucks with original injection pump seals failed due to swelling after some months of converting to biodiesel (22). However, the use of B20 in vehicles has logged over 50 million miles with few precautions and changes compared to diesel (24).

6.1.2.7. Piston

Deposits formed by the use of biodiesel are typically easier to clean compared to petroleum diesel (35). This is due to the solvency and absence of high molecular mass aromatics, which tend to form harder carbonaceous deposits.

6.1.2.8. Power/Torque

A benefit of the "fuel-oxygen-content" is that the engine's effective capacity and hence power is increased. A diesel engine runs lean and typically has an air: fuel ratio of 17 when operating at maximum power, or maximum fuel supply. The power is increased by injecting more fuel as a finely atomized spray to get intimate air contact. At higher fuel injection rates power stops increasing as air contact and mixing with fuel that supports combustion becomes mass transfer limited. This is due to limited time of contact despite ever better injection spray patterns. Since air contains about 21% oxygen, the oxygen: fuel ratio is about 3.6. The 11% oxygen thus increases the oxygen: fuel ratio to about 4.2 $[(3.6+0.11)/0.89]$. As oxygen supply is the limit of engine capacity or power output, theoretically the effective power achieved may be able to be increased when using B100 in place of petroleum diesel. This means that biodiesel may be effective in racing diesel engines, similar to the way in which methanol adds power in specially

47

optimized spark ignition engines. However, the greater percentage of heavy molecules in biodiesel, coupled with the higher viscosity and potentially larger droplets counter this factor. In practice, typical diesel engines are not optimized for biodiesel, so little, no or even negative power increases could be found.

Energy efficiency is the percentage of the fuel's thermal energy that is delivered as engine output. The heat and hence volume of combusted gas products is the most critical factor, but is countered by exhaust gas maximum temperature limits. Fuels with higher hydrogen content result in more molecules of product gases. In the case of biodiesel, the factor is less significant and varies depending on which diesel it is being compared to. For instance, paraffinic FT type diesels contain more hydrogen than aromatic containing crude oil derived diesels. The overall effect also depends on engine and operating condition, so more work is needed. Single claims, such as that biodiesel increases engine efficiency by a few percent, or decreases engine efficiency by some percentage should be treated with caution.

6.1.2.9. Injection Timing

Retarding the injection timing by 2-3 degrees can help overcome the effect of biodiesel's higher cetane number. The engine runs quieter and the fuel burns cooler, reducing nitrogen oxides (NO_x) emissions (22,28). However, doing so might invalidate engine certification, as it would be considered as tampering with the engine. Hence, engine/vehicle manufacturers do not recommend such modifications post sales. Another factor may be that biodiesel's higher bulk modulus (density change is lower with pressure) results in injection timing being advanced in pump-line-nozzle and unit pump type injection systems (as opposed to common rail, the dominant technology in light duty diesel engines). This is perhaps a more likely reason for retarded injection timing having a positive effect.

6.1.3. Emissions

Emission reports regarding biodiesel and ultra low sulfur diesel (less than 50 ppm) blends are inconsistent in their findings. There seems to be a link between emissions and duty-cycle, type of engine, and condition of the engine. However, there are as yet no known issues with any after-treatment systems being adversely affected by the use of biodiesel. The U.S. EPA has surveyed a large body of biodiesel emissions studies and the results are shown in Table 9.

Most biodiesel emission studies have been carried out on existing heavy-duty highway engines. The effects of biodiesel on emissions from heavy diesel engines meeting EPA's stringent Tier II emissions standards (introduced since model year 2007) have not been determined, and the EPA has concluded that the results of biodiesel tests in heavy-duty vehicles cannot be generalized to light-duty diesel vehicles or off-highway diesel engines. The work continues, but this should not stop the general growth of biodiesel as a substitute for petroleum diesel, as the CO₂ (GHG) emission benefits are clear and desirable and the inherent cleaner chemical composition including oxygen in the fuel molecules, will tend to reduce HC and PM emissions. Of course, urban areas with very high diesel consumption and air quality issues should follow proper impact

assessments when changing fuel types and characteristics. Excluding NO_x, the switch from diesels to biodiesel, whether as B100 or as blends, is likely to be positive, or at worst neutral.

Table 9: Average Biodiesel Emissions Compared to Diesel

Emission Type	B100	B20
Regulated		
Total Unburned Hydrocarbons	-67%	-20%
Carbon Monoxide	-48%	-12%
Particulate Matter	-47%	-12%
NO _x	+10%	+2% to -2%
Non-Regulated		
Sulfates	-100%	-20%*
PAH**	-80%	-13%
nPAH**	-90%	-50%***
Ozone potential of speciated HC	-50%	-10%

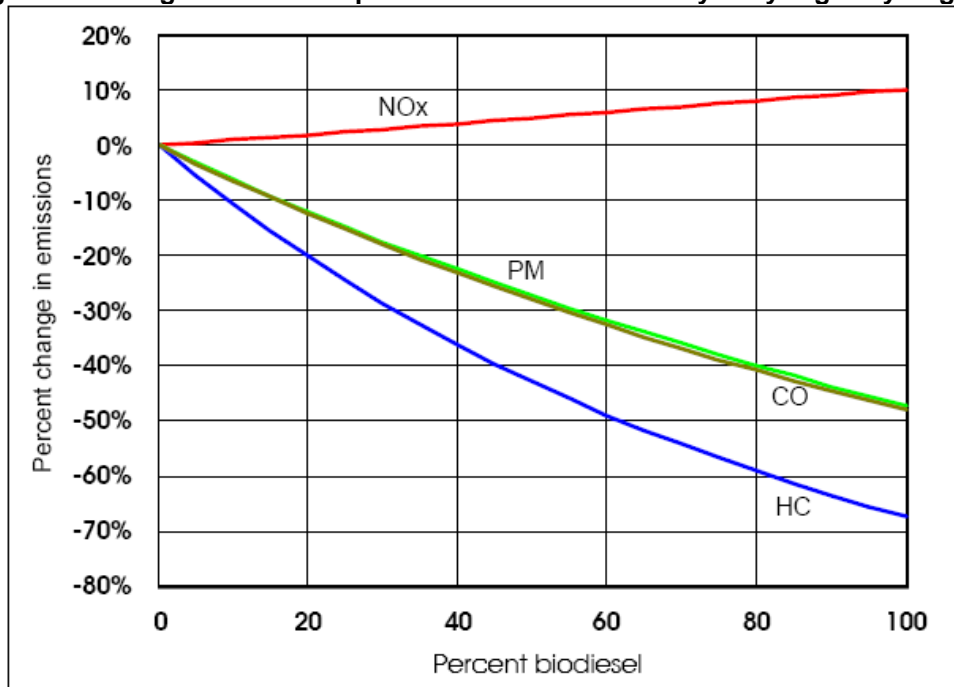
* Estimated from B100 result

** Average reduction across all compounds measured

*** 2-nitroflourine results were within test method variability

Source: U.S. EPA (41)

Figure 6: Average Emission Impacts of Biodiesel for Heavy-Duty Highway Engines

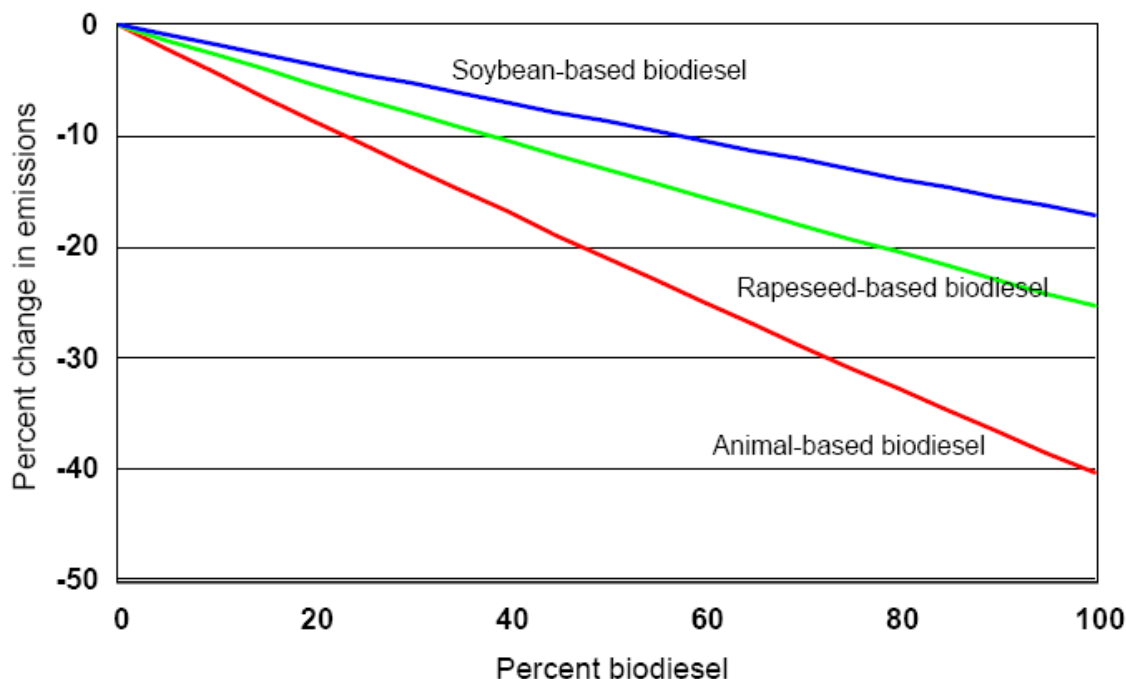


Source: U.S. EPA (41)

6.1.3.1. Carbon Monoxide

The exhaust emissions of carbon monoxide (CO) from biodiesel are on average 48% lower than those from diesel (41,4,28,42,43,44,45). CO emissions from palm oil based biodiesel were greater than those from jatropha (35). This is due to the higher density and lower oxygen content of the palm biodiesel.

Figure 7: Biodiesel Source Effect on CO for Heavy-duty Diesel Engines



Source: U.S. EPA (41)

6.1.3.2. Hydrocarbons

The oxygen in the biodiesel improves combustion and reduces hydrocarbon (HC) emissions (46). Also, biodiesel does not contain the heavy aromatics found in petroleum diesels, and this further reduces the HC and PM emissions, as they correlate with heavy (higher boiling point) aromatics (typically polynuclear aromatics). This is the reason for a PNA limit for EN 590 diesel. The exhaust emissions of total hydrocarbons (a contributing factor in the localized formation of smog and ozone) are on average 67% lower for biodiesel than diesel fuel (41,4,28,42,43,45).

6.1.3.3. Nitrogen Oxides

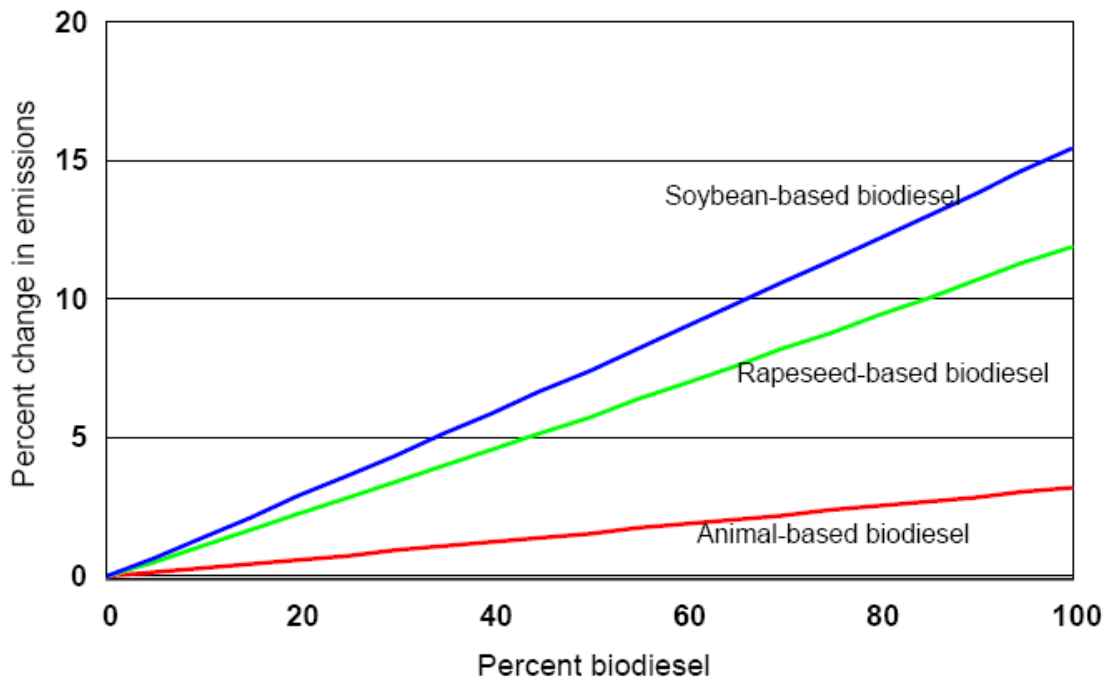
The effect on NO_x , the other main criteria pollutant emission from diesel engines, is less clear. The added oxygen and resulting enleanment causes increased NO_x formation. Further, the typical higher temperatures of leaner combustion as well as the oxygen chemical reaction favor increased NO_x formation.

The NO_x emissions increase or decrease depending on the engine family, testing procedures and feedstock. An inverse relationship with iodine value has been

associated with NO_x emissions (19). Cetane improvers could also help reduce NO_x emissions (19,47). However, the lack of sulfur in certain biodiesel allows the use of NO_x control technologies that can only be used with ULSD. Additionally, some companies have successfully developed additives to reduce NO_x emissions in biodiesel blends (4,7,28,42). However, care must be taken while using such additives as they could increase PM (47). Jatropha based biodiesel emits more NO_x than palm oil based biodiesel (35) (largely due to the greater relative oxygen content). Another strategy for reducing NO_x is blending with low aromatic diesel, kerosene or FT diesel (47).

Therefore, the effect on NO_x varies (48,49). Conflicting results on NO_x prompted the EPA to note in its regulatory impact analysis (50) for the Renewable Fuel Standard that significant new testing is planned to better assess the impact of biodiesel on NO_x and other exhaust emissions from the in-use fleet of diesel engines. Of course, adjusting the engine settings, such as injection timing can change the NO_x emissions. In Europe higher NO_x may provide a challenge for OBD (on-board diagnostic) systems (now being implemented), as these use a NO_x sensor in the exhaust.

Figure 8: Biodiesel Source Effect on NO_x for Heavy-duty Diesel Engines



Source: U.S. EPA (41)

6.1.3.4. Ozone

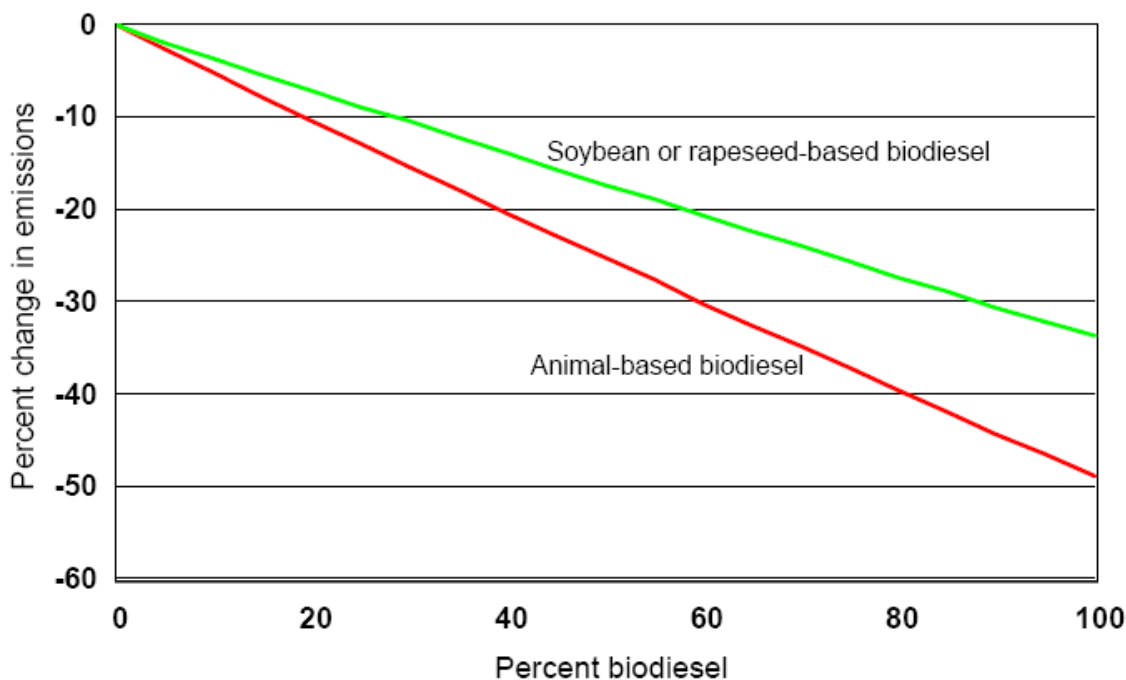
The ozone (smog) forming potential of biodiesel hydrocarbons is less than diesel fuel. Speciated hydrocarbon emissions are 50% less than measured for diesel fuel (42).

6.1.3.5. Particulate Matter

Based on the higher density, biodiesel injects about 6% more mass of fuel, so all else equal, this would increase the fuel: air ratio and increase PM emissions. However, as

biodiesel contains 11% oxygen, it improves combustion (makes combustion more complete and cleaner), and reduces PM emissions (46). Biodiesel exhaust emissions of PM are about 47% lower than diesel. For B20, this was reduced by 12% compared to diesel (41,4,28,42,43,45).

Figure 9: Biodiesel Source Effect on PM for Heavy-duty Diesel Engines



Source: U.S. EPA (41)

6.1.3.6. Polycyclic Aromatic Hydrocarbons

Biodiesel emissions show decreased levels of polycyclic aromatic hydrocarbons (PAH) and nitrated polycyclic aromatic hydrocarbons (nPAH), which have been identified as potential cancer causing compounds. In Health Effects Testing, PAH compounds were reduced by 75% to 85%, with the exception of benzo(a)anthracene, which was reduced by roughly 50%. Targeted nPAH compounds were also reduced dramatically with biodiesel, with 2-nitrofluorene and 1-nitropyrene reduced by 90%, and the rest of the nPAH compounds reduced to trace levels (42). This is to be expected based on the absence of precursor aromatics in biodiesel, and particularly polynuclear aromatics.

Reductions of smoke density when fueling with biodiesel and B20 as compared to No. 2 diesel have been reported (51,52,53,54,55). One study used sunflower derived biodiesel (51), another used rapeseed biodiesel (52), while the other two studies used soybean biodiesel (53,54,55). However, increase in smoke density was observed when using karanja-based biodiesel (56). There were noted reductions in HC and CO emissions with biodiesels (53,54,57,58,59). A study noted increases in HC and CO, and reductions in NO_x emissions (60). This appears to be an anomaly, and may be vehicle technology and settings dependent (28).

6.1.3.7. Sulfur

Biodiesel, apart from fertilizer contamination in the oils and sulfuric acid process washing, is essentially sulfur free and therefore acts as an exhaust emissions system technology enabler. It typically helps reduce the sulfur content directly in proportion to its blend ratio and the respective sulfur contents, and thus helps to improve catalyst efficiency. SO_x emissions are consequently almost eliminated with pure biodiesel (28,42).

6.1.3.8. Catalyst Deactivation

Some elements are present in biodiesel that are known to be responsible for catalyst deactivation, including phosphorus from the feedstock, and several metals that may be used in the process (Na, K – from the hydroxide; Ca, Mg- from water washing). Concerns with catalyst deactivation and ash accumulation in after-treatment systems of heavy-duty engines are accentuated by the volumes of fuel consumed and the more demanding durability requirements when compared to light-duty vehicles (61). A benefit is that the character of biodiesel soot results in lower temperatures needed to regenerate PM traps, in some cases eliminating the need for active regeneration (the use of a discrete fuel injection event to raise the temperature to burn off the soot). That in turn can positively affect fuel economy, and potentially the design of after-treatment systems.

6.1.3.9. Diesel Particulate Filter Systems

Laboratory studies have shown that blending biodiesel with diesel fuel can lower the DPF balance temperature. Faced with engine durability issues and instances of DPF clogging, several manufacturers disallowed the use of neat biodiesel in Euro III and later diesel passenger cars equipped with active DPF systems (61).

6.2. Gaps and Uncertainties

Most of the vehicle, engine and fuel component research and experience has been with rapeseed and soy based biodiesel. The majority of the research conducted has been on heavy-duty vehicles, such as trucks and buses, being the sector that consumes the most diesel fuel. The majority of problems reported with the use of low-level biofuel blends have been attributed to off-spec biodiesel and/or improper blending leading to fuel separation. The field trials have been largely restrictive due to the unavailability of biodiesel at the consumer level. Those fleets where tests were conducted were mainly captive fleets, and not always representative of the vehicle parc and its diverse operating conditions. However, many municipal vehicle fleets in France, including light duty and passenger vehicles, have been operating under wide-ranging conditions on B30 for many years with satisfactory results.

6.3. Possible Problems and Acceptance

Given the uncertainties, it is perhaps advisable to look at what specifications are desired

to avoid or minimize problems. This is examined in Table 10 below.

Table 10: Properties of European Rapeseed Biodiesel That Can Cause Vehicle Problems

Property/Contaminant	Effect	Components affected
Fatty acid	Increases corrosion rate of zinc Forms salts	Fuel system
Fatty acid methyl esters	Solvent effect	Fuel system elastomers
Glycerol Mono, Di and Tri glyceride	Corrodes non-ferrous metals Blocking filters and coke deposits	Fuel system
Methanol	Corrosion of aluminium and zinc Low flash point	Fuel system, Safety, as toxic and flammable
Modulus of elasticity	Increases injection pressure	Fuel system pressure leaks
Alkaline and Alkaline earth (Na + K, Ca + Mg)	Increases corrosion rate of non- ferrous metals. Increase in sludge & deposit build- up.	Fuel system, Emissions via harming after-treatment devices
Solid impurities	Lubricity (wear) problems	Fuel system, Engine
Viscosity	Fuel flow problems. Increased stress on components Causes component heating Injection spray problems	Fuel system
Water	Hydrolysis of FAME to fatty acid and methanol Corrosion Bacterial growth Electrical conductivity	Fuel system
CFPP	Affects flow rate of fuel through the filter and injectors due to crystallization and gelling. Can cause pumps to cavitate. Vehicle starting problems in cold temperatures through crystallization	Fuel system
Total contamination	Machine standstill through filter backfill, potential consequential damage to the injection pump as a result of insufficient lubrication / cooling by circulating fuel.	Fuel system
Iodine Number	Inverse relationship with NO _x emissions. Affects oxidation stability and CFPP	Emissions
Residual coke	Injection pump and piston ring deposits	Engine
Ash	Engine and exhaust contamination	Emissions, Engine
Oxidation Stability	Precipitation of polymers Acid formation and fuel system attack &/or deposits	Fuel system, Engine
Acid number	Corrosion	Fuel system

Source: Bio-Diesel Quality Management Work Group

Biodiesel meeting the U.S. ASTM or the EU specs when used as a 5 vol% blend with conventional diesel is not expected to cause any problems with vehicles and has been successfully introduced seamlessly without any labeling requirements in various economies worldwide. This blend level is also approved by FIE manufacturers. Most APEC economies are supplying diesel with 500 ppm sulfur or lower, thus vehicles are expected to have components which are compatible with B5.

Blends of biodiesel with more than 5 vol% but less than 20 vol% could have some engine and fuel system compatibility issues. Most of these issues can be rectified with minimal maintenance and replacement of parts. No critical failure is expected without warning to the operator in the form of reduced power and starting problems. Currently, very few manufacturers in limited numbers of their product lines have certified vehicles for use with these blend levels. It is expected that the availability of such blends would require additional labeling requirements and consumer awareness of possible compatibility issues. However, most new vehicles sold today are expected to be compatible, with problems, if any, arising in old vehicles.

Blends above B20 are currently not recommended by most manufacturers. There is limited data available on compatibility and performance related issues with these blends over the useful vehicle life. These blends will require biodiesel compatible equipment and modifications to engine timing to maintain performance. This issue can also arise in low-level blends of biodiesel if improper blending practices result in fuel separation, such that the vehicle will be using B100, which has separated from the diesel.

During the recent past with high economic growth rates, and associated production, sales and construction booms, engine and vehicle manufacturers have taken a more measured approach to supporting biodiesel in their diesel-engine vehicles. Engine manufacturers have two primary areas of concern:

1. stability and quality of existing biodiesel; and
2. effect of biodiesel combustion on the emissions and performance of their existing engines, and on the emerging advanced combustion regime engines they are designing to meet more stringent emissions requirements.

Both concerns stem directly from the basic chemistry of biodiesel and its production process. Problems specifically caused by any fuel, diesel, biodiesel or their blends, are not considered manufacturing defects and will not be covered by any vehicle, engine or FIE manufacturers warranty. The warranty covers materials and workmanship.

6.4. Relevance for APEC Vehicles

It appears that B5 blends of biodiesel complying with a suitable specification will not cause many problems and can be relatively aggressively adopted.

Higher levels up to B20 should cause only limited problems, but it is nevertheless recommended that the introduction thereof is gradual and preferably in dedicated fleets that are well maintained and monitored.

A similar approach is recommended for B100 use in vehicles. This needs to be

deliberate choice by the user, based on aspects, such as:

- the fuel manufacturer/supplier agreement and support provided,
- quality control,
- market testing or experience, and
- fleet type, age and operation.

7. APEC Biodiesel Standard

7.1. Approach

The approach to setting a common biodiesel standard can vary as follows:

1. Requiring diesel blends that contain biodiesel to comply with:
 - a. Applicable diesel specification – This raises further issue as to what extent are APEC or national or global diesel specs aligned. There is a convergence occurring, if with some lag, due to globalization;
 - b. Applicable biodiesel specifications, possibly with certain waivers provided to enable use of varying biodiesels, at varying treat rates; and
 - c. A new biodiesel blend standard – This is as suggested for B10 in Europe and B20 in the U.S., although it includes the caveat that the biodiesel must comply with the EN 14214 and ASTM D6751 standards respectively. The informal B30 standard in France requires EN 14214 compliant biodiesel to be blended.
2. Establishing a B100 standard that can:
 - a. Ensure successful use in the market as B100. This is the European approach using EN 14214. This is vehicle dependent, and blends higher than B5 or B20 are usually not available to the public and are predominantly supplied to captive fleets and niche markets;
 - b. Ensure a satisfactory product when blended with on specification mineral diesel. The blend rate limits would be set, i.e. B2, B5 as is the current European case; B10 and B20, the current U.S case; and
 - c. Provide a biodiesel blend component that meets an agreed quality standard and has known characteristics, so that it can be blended with other biodiesel components, and/or additives and/or a blendstock diesel resulting in a finished fuel blend that complies with the applicable diesel specifications. This represents the Brazilian approach, which requires optimization of the blend, including blend rates of the biodiesel (and other components), and certifying/testing of the resultant blend.

The above six options are not exclusive and are tabled as options to facilitate further discussion amongst APEC stakeholders.

The following positions are assumed:

- Given that the focus is on providing a specification for manufacturing and trading biodiesel amongst APEC economies, a B99 or B100 standard is desired for biodiesel to be used as a blend component ;
- Given that acceptance by vehicle manufacturers and no problems for customers is desired, coupled with the apparent intent (also linked to production capacity role out) to start at relatively low-levels of blends in APEC economies, it is

assumed the biodiesel will be used initially in B5 blends that should comply to the applicable diesel standard; and

- Given that biodiesel manufacturers will prefer a single standard, it is further assumed that the biodiesel blendstock for B5 will also be the B100 specification, and the same biodiesel can be blended at higher levels, such as B10, B20 and B50.
 - These higher blends may require waivers from the conventional diesel specification, or may require special standards. In practice, blenders may maximize the biodiesel and vary the mineral diesel blendstock and still comply with the applicable diesel specification (Ideally, both the biodiesel and diesel blend components should however comply with their individual specifications). This allows optimization of blending. In such cases, due to being tailored to specific user needs, for captive fleets, or where the higher blend of B100 is sold to the public, this should include specific communication such as product labeling.

7.2. Specification Parameters

The Brazilian performance based characterization forms a sound basis for further discussion, while including the European and ASTM requirements and examining current APEC national specifications..

Two main aspects need to be considered, as outlined in Table 11 below. These are:

1. applicability (and/or desirability) for APEC member economies, and
2. if desirable/applicable, then what should the limit(s) be.

The applicability must be guided by vehicle performance and durability requirements, including current and future emissions and durability of emission control devices.

The properties are also classified as to whether they are process or feedstock dependent.

- for process dependent properties, the limits indicated are based on current APEC national specifications, and
- for feedstock dependent properties, limits that restrict feedstocks should not be accepted unless there is a valid performance reason. Simply that they control that a certain feedstock is only used is not sufficient reason to include the limits.

Table 11 also proposes what testing frequency may make sense in practice, based on the Brazilian approach. It also refers to quality control testing by German biodiesel producers. This is not an immediate output, but rather follows the standard in place and includes meaningful statistical testing.

This table and structured approach is designed to facilitate meaningful discussion by APEC experts and stakeholders on the respective biodiesel performance properties.

Table 11: Brazilian Approach to Biodiesel Specifications, Adding European and ASTM and APEC National Specification Requirements to Facilitate Discussion of Parameters for APEC Standard

	Applied Frequency of testing in Brazil	Applicable to APEC	Most commonly noted limits in APEC Economies <i>NR=Not Reported</i>
Process Dependent			
Visual appearance	Every batch	Yes This limits more costly tests being done unnecessarily	Clear and bright No sediments, precipitates or residues
Water, or Water & sediment	Every batch	Yes Can be removed before marketing or blending, but not advisable as biodegradation and corrosion would occur in the water present while stored.	500 ppm Water and sediment more strict and common in APEC. Total contamination also addresses sediment.
Total contamination	Report; 3 monthly	Can be addressed by handling, filtration etc. Depends on national practices and diesel standard.	Could be addressed by specific customer and market agreed parameters
Acidity	Every batch	Yes Means of checking for control of acid removal and conversion of organic acids to ester. Indicates the amount of free fatty acids, that are natural degradation products from oils and fats; high number indicates a manufacturing problem and/or excessive oxidative degradation. Acids may cause corrosion and degradation and lead to filter & injector blockage, engine deposits and less durability.	0.5 mg – 0.8 mg KOH/g 0.5 should be achievable by proper process and controls
Copper corrosion	Every batch	Yes	Class 1 – 3 Class 1 should be achievable
Flash point	Every batch	Yes	100°C –120°C Propose 100°C to include coconut oil. This limit way exceeds diesel specifications.
Methanol content	Every batch	Covered by flash point	NR ⁽¹⁹⁾ – 0.2% Probably not required with flash point of 100°C minimum

¹⁹ NR = no requirement

	Applied Frequency of testing in Brazil	Applicable to APEC	Most commonly noted limits in APEC Economies <i>NR=Not Reported</i>
Free glycerol/glycerin Total glycerol/glycerin	Every batch	Yes Measures the amount of unconverted or partially converted fats and oils as well as byproduct glycerin in the fuel. Excess levels lead to gumming that results in deposits and filter blockage.	0.02% m/m 0.24% m/m Common agreement to include this requirement
Ester content, min	Report; 3 monthly	ASTM does not include. Requires expensive laboratory equipment and the reproducibility is poor. Many large biodiesel producers in Germany do not have this apparatus and do not test on every batch.	NR – 96.5% No requirement for every batch, but plants should establish typicals, as some customers would need value.
Mono-, di-, triglycerides		ASTM does not include. Covered by Free and Total Glycerides. German plants do not test regularly.	Most NR Not required prior to Euro IV & V advanced vehicle emissions control systems.
Na & K, max	Every batch	Yes Actual testing would depend on which of these hydroxides is used in a respective biodiesel plant. Some specifications require monitoring less frequently than every production lot but more frequently than every six months	NR, report, 5 ppm Establish baseline for a process and then guarantee. Use external lab testing to confirm periodically.
Ca & Mg, max	Report by testing 3 monthly	Yes May be tested monthly as part of production lot testing	NR, report, 5 ppm Easy to test, with Na & K. Establish baseline for a process and then guarantee. Test depending on justified frequency

	Applied Frequency of testing in Brazil	Applicable to APEC	Most commonly noted limits in APEC Economies NR=Not Reported
Phosphorous, max		Probably Yes Depends on vehicle emission control devices and thus also on blends. Most APEC economies at Euro III until 2008, and Euro IV from 2012, so will require by 2012	NR, 10 max more common Establish baseline for a process and feedstock and then guarantee. Test depending on justified frequency testing
Carbon residue	Every batch	Yes	0.05% m/m Include limit
Sulfated ash		Yes Measures the amount of metals, typically the residual alkali catalyst present in the biodiesel as well as any other ash forming compounds that could contribute to injector deposits or filter reduced life.	0.02% m/m Include limit
Raw Material Dependent (Feedstock)			
Density; viscosity; CFPP	No limits for biodiesel alone, apart from logistics requirements depends on additive and blending	Yes Can address with additives or blending	Report and guarantee Actual limits depend on regional & blend requirements, as well as fuel handling and vehicle fuel systems
90% distillation, max	Every batch	Yes	NR – 360°C Establish baseline for a process and feedstock and then guarantee
Cetane number	Report by testing 3 monthly	Yes Typically just test 6 monthly or when change made to process and/or feedstock	Align with diesel and depends on blend use Establish baseline for feedstock (& process) and then guarantee

	Applied Frequency of testing in Brazil	Applicable to APEC	Most commonly noted limits in APEC Economies NR=Not Reported
Iodine number	Report; 3 monthly	Yes, until a more suitable test establishing biodiesel stability is determined.	Maximum limit depends on feedstock and may be able to be addressed by additives. Show why not needed to test based on feedstock and plant data, coupled with use of anti-oxidant
Oxidation stability	Every batch	Yes Test at 110°C must typically pass for 6 hours, could be 10 hours based on work done in Japan	Should be able to address increment from 6 hours to 10 hours with additives. More work is needed and being conducted internationally.
Linolenic acid methyl ester content	No limits for Brazil and ASTM; only EN	OEMs insist this is most important	Limits feedstock
Polyunsaturated methyl esters		No	Limits feedstock
Both Process and Feedstock Dependent			
Sulfur	Report; 3 monthly tests	Yes Sulfur leads to emissions and may negatively effect exhaust catalysts. Normally a regulated requirement. Standard grade limit of 500 ppm is common. All biodiesel should easily comply. Where regulated stricter, such as 50 ppm max, this would be the requirement.	Align with diesel; Can sometimes blend down, with other biodiesels or with low sulfur conventional diesels. Establish baseline for a process and feedstock and then guarantee

The limits currently in use in APEC economies were reviewed at the APEC Thailand Workshop on October 25th and 26th, 2007 by categorizing them as follows:

1. Regulatory and emission requirements
2. Engine and emissions control system performance
3. Measure of direct performance, as regards usability and durability
4. Indirect Durability / Usability, or inferred measure of performance by specifying chemical limits. These can limit certain feedstocks, and should be replaced by performance based specifications once suitable parameters and test methods have been defined.

The results of the Thailand workshop preliminary discussion of current biodiesel specifications are presented in Table 12 below.

Table 12: APEC Biodiesel Quality Standard and Harmonization Initiative

	ASTM D6751	EN 14214	Typical APEC Economy	Discussion & Conclusions
Regulatory and Emissions				
Max Sulfur (ppm)	15 / 500	10	10 - 500	Regulatory requirement per economy Buyer-seller specified
Min Flash Point (°C)	130 / 93	120	93 - 130	For non-hazardous classification in U.S. min of 93°C is required. To show methanol controlled certify at >130°C
Max T90 Distillation (°C)	360	-	The Philippines, Australia Indonesia @ 360 °C	Other (performance) tests control contaminants Biodiesel reduces PM and HC emissions so test not required for emissions reasons
Engine and Aftertreatment Performance				
Cetane Number	47	51	47 - 51	Higher than 47 (EN) is required for emissions. This is based on diesel tests, so not necessarily applicable to biodiesel. Higher minimum ambient temperatures reduce start-up emissions. Blending not necessarily linear Additives can be used
Min-Max Density @15 °C, kg/m ³	-	860 – 900	820 - 900	Agreement
Min-Max Viscosity @ 40 °C, cSt	1.9 – 6.0	3.5 – 5.0	1.9 – 3.5 min 4.5 – 6.0 max	Coconut below 3.5, and tallow and palm can exceed 5 Requirement should be for the final blend

	ASTM D6751	EN 14214	Typical APEC Economy	Discussion & Conclusions
Max phosphorus ppm	10	10	10 China no spec	Agreed
Max Alkali metals (Na + K), ppm	5	5	No spec, report, 5	Depends on after-treatment
Max Ca + Mg, ppm	5	5	No spec, report or 5	Depends on after treatment
Max CFPP, °C	–	+5 to - 44	No spec Chinese Taipei =0 Indonesia = +18	Requirement should be for the blend
Max cloud point °C	Report	-	No spec The Philippines = Report	Not needed
Direct Usability and/or Durability				
Max CCR 10%, wt%	-	0.3	0.1 – 0.3 The Philippines no spec	Difficult for biodiesel to fractionate 10 %, so not recommended.
Max CCR 100%, wt%	0.05	-	Korea, Chinese Taipei, Thailand, Japan, China =no spec	Recommended to use
Max water and sediment , vol%	0.05	-	Japan, NZ, Chinese Taipei, Thailand, China =no spec	Agreed to replace by separate testing
Max water, ppm	-	500	Australia, Indonesia, The Philippines, Korea = no spec	Agreed
Max Ash, wt%	0.02	0.02	0.01 – 0.02	Agreed at 0.02 May reduce later
Total Contamination, ppm	-	24	China, Indonesia, The Philippines, Korea = no spec	Agreed

	ASTM D6751	EN 14214	Typical APEC Economy	Discussion & Conclusions
Max Cu corrosion, 3 hr at 50 °C	3	1	1 Indonesia, The Philippines = 3	Needs further work for alignment. In practice biodiesel complies easily
Max Methanol content, wt%	0.2	0.2	China, The Philippines = no spec	Agreed
Max free glycerine, wt%	0.02	0.02	Korea = no spec	Agreed
Min oxidation stability @ 110 °C, hrs	3	6	No spec, 3 or 6 Japan = 10	Needs further work
Max total glycerin, wt%	0.24	0.25	0.24 – 0.25	Agreed and 0.24 recommended
Indirect (Derived) Usability and/or Durability				
Min Ester Content	-	96.5	China, The Philippines, US = No spec	Method developed for RME, so does not show lower molecular weight from CME
Max non-ester	-	None except additives	No spec	Agreed to exclude
Max acid value	0.5	0.5	0.5 – 0.8	Simple test. Agreed to include. Limit not agreed.
Max glycerides – mono, di, tri	-	0.8; 0.2; 0.2	No spec	No agreement. Prefer direct tests of performance
Max linolenic acid methyl ester	-	12	Australia, China, Indonesia, The Philippines, Korea, U.S. = No spec	Limits certain feedstocks with no clear reason. No agreement. Prefer direct tests of performance
Max polyunsaturated methyl ester	-	1	No Spec, Chinese Taipei = 1	No agreement. Prefer direct tests of performance

	ASTM D6751	EN 14214	Typical APEC Economy	Discussion & Conclusions
Max iodine number	-	120	115 - 120 Australia, China, The Philippines, Korea, U.S. = No spec	Limits certain feedstocks without certain reason. Max limit of 130 preferred. No agreement. Prefer direct tests of performance
Mandated detergents & additives	-	-	-	No agreement. Further discussion required

8. Conclusions

Biodiesel trade between economies is likely to be limited, as even in economies such as Malaysia that have major capacity, the consumption of diesel is so high that captive use may take much of production for some years to come.

Nevertheless, it is important for the development of the biodiesel industry and its ultimate success that there is alignment of specifications, so that where trade is possible, it can occur without technical barriers. A challenge for APEC member economies is that many have Euro 2 and 3 equivalent vehicles, while others such as the United States and Japan have Tier 2/ Euro 4 type vehicles with sophisticated on-board diagnostics equipment and sensitive catalytic emissions control devices. Many economies have fully equipped laboratories, while several do not.

A standard provides a basis on which to manage consumer acceptance by ensuring vehicle compatibility. It also provides a clear target for production plants, particularly those located in high capacity feedstock regions that are able to take advantage of economies of scale to produce biodiesel for export.

This position paper has highlighted factors that effect biodiesel properties and in turn how these properties effect performance in vehicles, including effects on exhaust emissions. The international work that is being carried out by the OEMs and by Brazil, the U.S. and Europe under a tripartite agreement on harmonization of biodiesel standards should provide further insights.

Fortunately, given the diversity of feedstocks that have been used and tested to date, the development of a feedstock neutral standard is now much easier. Nevertheless, careful examination of required diesel fuel properties and the associated limits, particularly for those properties that are feedstock dependent, would be required, as otherwise the biodiesel industry may be unnecessarily constrained. Much of the data that has been generated to date is based on RME or SME, and PME tested in Euro 0 to Euro 2 vehicles. Further research work including alky esters produced from new feedstocks such as algae and jatropha should be undertaken.

Expert stakeholders from the APEC economies will also need to:

- conduct an assessment of testing facilities and laboratories in member economies. The representative from Indonesia mentioned for example that there is no laboratory in Indonesia capable of testing all the biodiesel parameters in the ASTM D6751 or EN 14214 standards;
- establish accredited test facilities for round-robin testing between APEC economies;
- review all available test data for feedstock dependant variables, and identify further research work required in support of performance based specifications;
- Include the FIE manufacturers in further discussion

APEC expert stakeholders are encouraged to examine and debate the data presented

in this report with a view to reaching consensus on the quality control parameters required for an acceptable performance based B100 biodiesel standard to be adopted by APEC member economies.

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10. Appendices

10.1. Global Biodiesel Specifications

Biodiesel	Australia	Brazil	China	Chinese Taipei	EU	India
	(1)	ANP Act No. 42 / 2004 (1)	GB/T 20828-2007	CNS 15072 K5155	EN 14214:2003 (1)	IS 15607:2005 (1)
Cetane Number, min	51	Report	49	51	51	51
Ester content, wt%, min	96.5	Report	–	96.5	96.5 (2)	96.5
Sulfur, ppm, max	10	Report	50	10	10	50
Density at 15°C, kg/m ³ , min-max	860 – 890	Report (2)	820 – 900 (1)	860 – 900	860 – 900	860 – 900
Viscosity at 40°C, cSt, min-max	3.5 – 5.0	Report	1.9 – 6.0	3.5 – 5.0	3.5 – 5.0	2.5 – 6.0
Flash point, °C, min	120	100	130	120	120	120 (2)
CCR, 100%, wt%, max	0.05	–	–	–	–	0.05
10%, wt%, max	0.3	0.1	0.3	0.3	0.3	–
Water and sediment, vol%, max	0.05	0.05	None	–	–	–
Water, ppm, max	–	–	500	500	500	500
Ash, wt%, max	0.02 (2)	0.02	0.02 (2)	0.02 (1)	0.02	0.02 (3)
Total contamination, ppm	24	Report	–	24	24	24
Copper corrosion (3hr at 50°C), max	Class 1	–	1	1 (2)	Class 1	Class 1
Acid value, mg KOH/g, max	0.8	0.8	0.8	0.5	0.5	0.5
Methanol, wt%, max	0.2	0.5 (3)	–	0.2	0.2	0.2 (4)
Monoglycerides, wt%, max	–	Report	–	0.8	0.8	–
Diglycerides, wt%, max	–	Report	–	0.2	0.2	–
Triglycerides, wt%, max	–	Report	–	0.2	0.2	–
Free glycerol, wt%, max	0.02	0.02	0.02	0.02	0.02	0.02
Total glycerol, wt%, max	0.25	0.38	0.24	0.25	0.25	0.25
Linolenic acid methyl ester, wt%, max	–	–	–	12	12	–
Polyunsaturated methyl ester, wt%, max	–	–	–	1	1	–
Iodine number, max	–	Report	–	120	120	Report
Phosphorus, ppm, max	10	Report	–	10	10	10
Alkali, (Na+K), ppm, max	5	10	–	5	5	Report
Metals, (Ca+Mg), ppm, max	5	Report	–	5	5	Report
Distillation T90, °C, max	360	360	–	–	–	–
CFPP, °C, max	–	–	Report	0 (3)	+5 to -44 (3)	–
Oxidation stability at 110°C, hr, min	6	6	6	6	6	6
Cloud Point °C	–	–	–	–	–	–

Biodiesel	Indonesia	Japan	Korea	New Zealand	The Philippines	Thailand	U.S.
	SNI 04-7182-2006 (1)	(1)	(1)	NZS 7500:2005 (1)	PNS 2020:2003 (1)	(1)	ASTM D6751-07b (1)
Cetane Number, min	51	51	(2)	51	42 (2)	51	47
Ester content, wt%, min	96.5	96.5	96.5	96.5 (2)	–	96.5	–
Sulfur, ppm, max	100	10	10	50 or 10 (3)	500	10	15 / 500
Density at 15°C, kg/m ³ , min-max	850 – 890	860 – 900	860 – 900	860 – 900	–	860 – 900	–
Viscosity at 40°C, cSt, min-max	2.3 – 6	3.5 – 5.0	1.9 – 5.0	2.0 – 6.0 (4)	2.0 – 4.5	3.5 – 5.0	1.9 – 6.0
Flash point, °C, min	100	120	120	100	100	120	93
CCR, 100%, wt%, max	0.05	–	0.1	0.05	0.05	–	0.050
10%, wt%, max	0.3	0.3	–	0.3	–	0.3	–
Water and sediment, vol%, max	0.05	–	0.05	–	0.05	–	0.050
Water, ppm, max	–	500	–	500	–	500	–
Ash, wt%, max	0.02 (2)	0.02 (2)	0.01	0.02 (5)	0.02 (3)	0.02 (2)	0.020020 (2)
Total contamination, ppm	–	24	–	24	–	24	–
Copper corrosion (3hr at 50°C), max	Class 3	Class 1	Class 1	Class 1	Class 3	Class 1	No. 3
Acid value, mg KOH/g, max	0.8	0.5	0.50	0.5	0.5	0.5	0.50
Methanol, wt%, max	–	0.2	0.2	0.2	–	0.2	0.2 vol%
Monoglycerides, wt%, max	–	0.8	–	0.8	–	0.8	–
Diglycerides, wt%, max	–	0.2	–	–	–	0.2	–
Triglycerides, wt%, max	–	0.2	–	–	–	0.2	–
Free glycerol, wt%, max	0.02	0.02	–	0.02	0.02 (2)	0.02	0.020
Total glycerol, wt%, max	0.24	0.25	0.24	0.24	0.24 (2)	0.25	0.240
Linolenic acid methyl ester, wt%, max	–	12	–	12	–	12	–
Polyunsaturated methyl ester, wt%, max	–	–	–	–	–	–	–
Iodine number, max	115	120	–	120 (6)	–	120	–
Phosphorus, ppm, max	10	10	10	10	10	10	10
Alkali, (Na+K), ppm, max	18	5	5	5	–	5	5
Metals, (Ca+Mg), ppm, max	–	5	5	5	–	5	5
Distillation T90, °C, max	360	–	–	–	360	–	360
Cold Filter Plugging Point, CFPP, °C, max	–	(3)	–	–	–	–	–
Oxidation stability at 110°C, hour, min	–	(4)	6	6	–	6	3
Cloud Point °C	–	–	–	–	Report	–	Report

Australia

1. Australia allows for any biodiesel blend, however it must meet the diesel specs under the Fuel Quality Standards Act 2000; and
2. Sulfated.

Source: Fuel Standards (Biodiesel) Determination 2003

Brazil

1. Brazil allows for 2 vol% max biodiesel limit. The biodiesel blend must meet Portaria ANP No. 310/2001 diesel specifications with amendments. Permission for higher vol% blends can be obtained from ANP for test fuels;
2. Measured at 20°C; and
3. MeOH or EtOH.

Source: ANP

China

1. Measured at 20°C; and
2. Sulfated ash.

Source: State Environmental Protection Administration

Chinese Taipei

1. Sulfated; and
2. At 100°C.

Source: China National Standard

EU

1. The EU allows for 5 vol% max biodiesel blends as long as they comply with the conventional diesel requirements under Directives 98/70/EC and 2003/17/EC and EN 590:2004. 100% biodiesel fuels are not regulated by these fuel quality requirements but must solely comply with EN 14214: 2003;
2. The addition of non-FAME components other than additives is not allowed; and
3. Depends on climate rating. This property is to be amended to be applicable to the finished blended fuel.

Source: EN 14214:2003

India

1. "Biodiesel (B100) Blend Stock for Diesel Fuel - Specification." The standard states that biodiesel is a fatty acid alkyl (methyl or ethyl) ester (FAME/FAEE) for use as a blend component of up to 20 vol% with diesel fuel;
2. Pensky Martens test method;
3. Sulfated; and
4. MeOH or EtOH; MeOH is applicable for fatty acid methyl ester. EtOH is applicable for fatty acid ethyl ester.

Source: IS 15607:2005

Indonesia

1. Up to 10 vol% biodiesel is allowed in diesel fuel; and
2. Sulfated.

Source: Agency for the Assessment and Application of Technology

Japan

1. Up to 5 vol% biodiesel is allowed in diesel fuel;
2. Sulfated; and
3. To meet diesel fuel specifications.
4. Based on mutual agreement between parties concerned.

Source: Ministry of Economy, Trade and Industry

Korea

1. Under the Petroleum and Petroleum Alternative Fuel Business Act, the maximum blending limit for biodiesel with conventional diesel fuel is set at 5 vol% (B5) since January 2006; 0.5 vol% max biodiesel was introduced by July 2006. Up to 20 vol% (B20) is allowed to be used in certain fleets such as large buses or trucks. All biodiesel blends must meet diesel specs under the act; and
2. No cetane number or index set for B100, however cetane number required at min 45 for B5 and B20 blends.
3. CFPP is applied for severe weather season (from November 15th to February 28th)

Source: Korea Institute of Petroleum Quality

New Zealand

1. This standard covers biodiesel specifications for the manufacture of pure biodiesel (B100). There are two additional biodiesel specifications for use as automotive fuel for compression ignition engines as retail biodiesel blends of up to 5 vol% (B5) and non-retail biodiesel blends of up to 100 vol% (B100);
2. The addition of non-biodiesel components to B100 other than additives is not allowed;
3. The standard is currently only voluntary thus a lower sulfur level than 50 ppm may be required in order to meet regulated sulfur limits for biodiesel;
4. In all cases blends shall meet the limit of 4.50 cSt. The viscosity of most fatty acid methyl esters made from commonly used feedstocks is expected to be less than 6.00 cSt. However, this limit may be increased to 7.50 cSt if it is required to include a wider range of feed-stocks or monoesters other than methyl esters;
5. Sulfated; and
6. Iodine values between 120 and 125 may be acceptable providing rigorous testing of cetane number, oxidation stability and linolenic acid limits has been conducted.

Source: NZS 7500:2005

The Philippines

1. Biofuels Act passed in January 2007. 1 vol% coconut methyl ester (CME) in diesel mandated for government vehicles started March 1, 2004;
2. Transition Standard; and
3. Sulfated.

Source: PNS2020:2003

Thailand

1. The current blend is divided into 2 grades which are 0-2 vol% and 5 vol% of biodiesel. Moreover, the blending grade B5 must meet the diesel specification and the oxidation stability is limited at 25 g/m³ (2.5 mg/100ml) max. Also, the government plans to use diesel B5 nationwide by 2011.
2. Sulfated

Source: Department of Energy Business, Ministry of Energy, Thailand

U.S.

1. Specification for B100 when used as a blend component with diesel

2. Sulfated

Source: ASTM

10.2. Feedstock Availability and Biofuels Potential in the APEC Region²⁰

Most Asian economies are significant agricultural producers and have excess production of commodities that could be used for biodiesel production, particularly palm and coconut. In fact, this region boasts the largest palm oil producers with Malaysia and Indonesia leading the world, thus creating a huge potential for biodiesel production. In addition, the Philippines is the world's largest coconut producer and exporter. China and India are the largest global ethanol producers behind Brazil and the U.S. Thailand is an agricultural-based economy that is able to grow various kinds of crops as feedstock to meet national and global biofuels demand. However, some economies (such as: Brunei; Hong Kong, China; Japan; Singapore; Korea²¹ and Chinese Taipei) would most likely depend on biofuels imports because of insufficient land to produce domestic feedstock for biofuels.

Compared to other regions, Asia boasts a wide variety of feedstocks. For biodiesel production, the current feedstocks mainly include waste cooking oil, palm, coconut, jatropha, rapeseed and tallow. Although second-generation biofuels are currently not prevalent in the region, economies such as China, Japan and New Zealand are already conducting trial biofuels production from wood, grass, algae and other biomass resources.

10.2.1. Australia

A wide range of feedstocks is used to produce biodiesel in Australia, including used cooking oil, canola oil, tallow and palm oil. Australia has set a target of 350 million litres of biofuels for the domestic transport fuel market in Australia by 2010. This will represent around 1% of the transport fuel market.

In support of this target, biofuels receive an effective excise exemption until 1 July 2011, after which effective excise will be applied in five equal steps with alternative fuels receiving a 50% discount on excise compared with conventional fuels. By 1 July 2015, the effective level of excise on ethanol will be 12.5 cents/liter compared with 38.143 cents/liter for petrol and 19.1 cents/liter for biodiesel compared with 38.143 cents/liter for diesel. However, recent amendments to fuel taxation arrangements have reduced the comparative level of assistance to biodiesel compared with diesel for business use of fuel.

Infrastructure grant programs have supported additional biofuels capacity and distribution. The Biofuels Capital Grants Program has allocated A\$ 37.6 million to new

²⁰ This section is based on Hart Energy Consulting Global Biofuels Center (www.ifqcbiofuels.org) data.

²¹ Based on the APEC accepted nomenclature. In this report 'Korea' refers to 'South Korea.'

biofuel capacity and the Ethanol Distribution Program has allocated A\$17.2 million to support the conversion of retail fuel sites to market E10.

Australia has also established a Biofuels Action Plan to monitor progress towards the 350 ML target. The Action Plan comprises individual Action Plans drawn up by the major oil companies, members of the Independent Petroleum Group, and the major retailers and outline details of expected sales and production of biofuels to 2010. Based on the most recent review of the Action Plan, Australia is on track to meet its target by 2010. In 2006, the total production of fuel ethanol in Australia was 62.7 ML. Biodiesel production in 2006, was 43.9 ML bringing total production of biofuels in 2006 to 106.6 ML compared with 28 ML in 2005.

Australia has also placed considerable emphasis on fuel quality standard and labeling issues with respect to biofuels. The Australian Government is working with the fuel and transport industry to establish biodiesel standards and on undertaking a trial of biodiesel's environmental properties (as a B5 blend) in Booderoo National Park. Australia has also commissioned a study on the health impact of ethanol to validate overseas research under Australian conditions.

10.2.2. China

There are a large number of potential feedstocks available in China for biodiesel production including waste cooking oil, cottonseed, Chinese pistachio, soybean, rapeseed, peanut, tallow and jatropha. In fact, China is home to more than 1,500 types of oil plants, with over 30 of them highly adaptable and widely distributed. China produces approximately 1.5 billion tons of agricultural and food wastes and residues each year, including 100 million tons of agricultural crop stalks. However, virgin oil feedstocks are not economically currently feasible for biodiesel production due to high cost. Because of the cooking and eating habits of locals, waste grease and oils are the existing feedstocks available in abundance for biodiesel. China consumes more edible oils than any economy in the world. In 2005 over 19.1 million tons of edible oils were consumed in China. Of these oils, about 35% goes into the drainage system and becomes waste grease and oils. About 3 million tons per year of waste oil and grease are estimated to be produced, of which potentially 2 million tons could be recovered and processed into biodiesel. Rapeseed is one promising oilseed for biodiesel production, with China ranked as the world's largest producer at 11.4 million tons in 2003, according to the United Nation's Food and Agricultural Organization (FAO). The majority (75%) of China's rapeseed production comes from the Yangtze Valley (8 million tons per year), which also is the largest continuous area growing rapeseed in the economy. In fact, over 29 million hectares of fallow land exist in the area of the Yangtze River that are suitable for potential rapeseed cultivation to produce a total of 21 billion liters/year of biodiesel. Still, some estimates indicate that lack of land upon which new crops could grow will make it more difficult to increase biodiesel production.

The Chinese government is cooperating with national oil companies CNPC and its subsidiary PetroChina as well as international organizations such as UNDP and Carbon

Positive to plant jatropha as a long-term feedstock solution in providing biodiesel. Jatropha will be mainly cultivated in the southwestern provinces of Sichuan, Yunnan and Guizhou. However, China does not have a policy framework specifically to support biodiesel as a fuel.

10.2.3. Chinese Taipei

The major feedstock for biodiesel is recycled cooking oil; potentially 45,000 tons/yr of used cooking oil can be collected for this purpose. Chinese Taipei has started to cultivate crops in order to produce biofuels as well as set up a biodiesel promotion plan. Starting from 2004, fleet trials of garbage trucks and public buses have been carried out using a wide range of blends (2% ~ 100%). Chinese Taipei aims to introduce B1 blends nationwide in 2008 and increase to B2 by 2010 in the fourth phase. Taoyuan and Chiayi counties began selling B1 blends on July 27, 2007, ahead of the nationwide B1 requirement. In order to achieve the target set by the government, they need biodiesel use to reach 100,000 kilo liters (26.4 million gallons) by 2010.

10.2.4. India

In India, non-edible oil is most suitable as biodiesel feedstock because the demand for edible oil exceeds the domestic supply. India has a potential of biodiesel production from non-edible oils, including Jatropha Curcas, Pongamia Pinnata, Pongamia Glabra, Madhuca Indica, Shorea Robusta, Mesua Ferra (Linn), Mallotus Philippines, Garcinia Indica and Salvadora. However, current utilization of non-edible oilseeds is very low. Jatropha is one of the most popular non-edible oilseeds used for biodiesel. However, it would take some time for jatropha plantations to become productive before the economy can realize its blending targets.

The government is looking to develop the biofuels program through the public- private partnership route promoting biofuel plantations on land owned by the government, communities and the private sector. Several local corporations have already started cultivating jatropha on wasteland or have begun contracting farming on private land.

Currently biodiesel is not blended with diesel fuel in the market on a commercial basis. The proposed National Policy on Biofuels is aiming for biofuel blending ratios of 5 vol% by 2012, 10 vol% by 2017 and increasing the blend to more than 10 vol% after 2017. The president has called for utilizing India's wastelands for the planting of jatropha and other biodiesel crops. As India has nearly 60 million hectares of wasteland, about 30 million hectares could be made available for plantations with an aim to produce a minimum of 2,272 liters/year/hectare of biodiesel.

10.2.5. Indonesia

As the second-largest palm oil producer and exporter in the world, Indonesia is looking to promote local biofuels use and set up a National Biofuel Development Team in 2006. A target has been set for 10% of petroleum demand to be replaced by biofuels in 2010. Since May 2006, national oil company Pertamina has been marketing blends up to B5

in phases in Jakarta and Surabaya. However, currently only 2.5 vol% of biodiesel is being blended due pricing concerns. *Jatropha* is also being looked at as an alternative feedstock.

10.2.6. Japan

Since gasoline is the major fuel consumed in the economy compared to diesel, biodiesel is not commercially widely available in Japan. Plans to introduce renewable fuels in Japan include the utilization of biomass energy. Starting 2010 Japan is aiming to introduce a program that recycles leftover food, livestock droppings and scrap wood as biomass energy to fuel vehicles, ships and power plants.

10.2.7. Korea

Although Korea²² has a lack of land to produce agricultural resources, the economy is actively promoting biodiesel through the use of tax exemptions and by requiring oil companies to supply diesel fuel blends containing 0.5 vol% biodiesel starting July 2006. Korea still does not have sufficient feedstock for biodiesel production and thus most feedstock is imported. Feedstocks used locally include waste cooking oil and soybean. Up to B20 is currently allowed for use in certain fleets such as large buses or trucks.

According to the Ministry of Commerce, Industry and Energy, Korea aims to increase its biodiesel blending ratio in diesel fuel from the current 0.5 vol% to 3 vol% by 2012. Previously, the government was planning to increase the blending ratio to 5 vol% starting July 2008 but has instead decided to increase gradually to B5 blending in the long run. The targets will be reviewed in the later half of 2010.

10.2.8. Malaysia

Malaysia is currently the world's largest palm oil producer and exporter, accounting for more than 80% of global production. It presents a unique case in producing biofuels mainly for export markets compared to other Asian economies. There is no biodiesel standard in place yet. However, according to initial measures to be implemented under the National Biofuel Policy, biodiesel standards and certification would be developed by Sirim Bhd, the national organization of standardization and quality. Malaysia plans to set the biodiesel blend at B5. For now, the MPOB has recommended that biodiesel produced must meet the European biodiesel standard EN 14214, especially seeing that much of this biodiesel is meant for export.

The government was aiming to expand the use of B5 in government and privately owned vehicles nationwide in 2007, before making its use mandatory in 2008.

²² Based on the APEC accepted nomenclature. In this report 'Korea' refers to 'South Korea.'

10.2.9. New Zealand

As part of their National Energy Efficiency and Conservation Strategy (NEECS), the government has set a voluntary target of 2 petajoules (PJ) or around 65 million liters of renewable transport fuel by 2012.

The government has agreed in principle to set a sales target of biofuels in law, also called the biofuels sales obligation, which would require oil companies to sell approximately 3.4 vol% of biofuels in the transportation sector by 2012. It is anticipated that the relevant law would be passed by parliament in or before 2008.

The current domestic biodiesel industry is very small approximately 15 million litres per year. The current feedstocks are wastecooking oil and tallow by using conventional transesterification process or the 1st generation technology. There is estimated to be 150,000 tonnes per year of tallow available which could be used to make approximately 150 million litres of biodiesel per year. Rapeseed is expected to be feedstock for biodiesel production from early 2008.

10.2.10. The Philippines

Being the leading coconut producing economy in the world with annual oil production of 1.4 billion liters (370 million gallons), the economy is aiming to supply CME for nationwide biodiesel blending. The Philippines is the only economy in Asia to mandate biofuels use by passing a Biofuels Act in January 2007. B1 was required starting May, 2007. In 2009, the blend will increase to B2. The Philippines also plants *Jatropha Curcas* and a March 2006 study by the Department of Science and Technology found 2 million hectares available for *Jatropha*, and this has the potential to produce about 11 billion tons/year of biodiesel.

10.2.11. Thailand

Palm oil is Thailand's main biodiesel feedstock. Waste cooking oil is used as feedstock as well. Thailand is also looking at *jatropha* as a long-term solution to produce biodiesel. The government promoted community-scale biodiesel production during 2004–2006 and set a nationwide B5 target by 2011 and B10 by 2012. Currently, biodiesel used in the country is categorized into 2 types, which are B2 and B5. As of 2007, biodiesel uses (both B2 and B5) in the country is on a voluntary basis. However, B2, will be mandated nationwide from April 1, 2008, with plans to mandate B5 by 2011.

10.2.12. U.S.

In the U.S., biodiesel is typically produced from soybean oil. The U.S. government has fostered the use of biodiesel through Energy Policy Act (EPAAct) requirements and tax incentives. EPAAct requires fleet operators to use a certain percentage of alternatively fueled vehicles (AFVs). The Energy Conservation Reauthorization Act of 1998, allows vehicle fleets that are required to purchase AFVs (e.g., government fleets) to generate credit toward this requirement by purchasing and using biodiesel.

Another driver is the U.S. Department of Agriculture grant program for biodiesel production through its Commodity Credit Corporation (CCC). Also, the Congress in Oct, 2004 passed a biodiesel tax incentive, structured as a federal excise tax credit, as part of the American Jobs Creation Act (JOBS Act) of 2004. The incentive is taken at the blender level. The tax credit under the JOBS Act was scheduled to expire at the end of 2006, but was extended in the energy legislation to the end of 2008.

The EPA Act of 2005 established the Renewable Fuels Standard (RFS) program, requiring increasing use of renewable fuels every year. The Energy Independence and Security Act of 2007 amended the RFS, requiring 34.07 billion liters in 2008, replacing the standard established in the EPA Act of 2005. The RFS requires biodiesel usage to reach 3.78 billion liters by 2012, starting with 1.89 billion gallons in 2009.

10.3. APEC Regional Experience with Production and Standards

10.3.1. Australia

In Australia there are a variety of feedstocks including tallow, canola, palm and waste cooking oils all being used and/or considered. Currently very limited support exists from diesel vehicle manufacturers for the use of diesel/biodiesel blends other than very low-levels (less than 5 vol%) in light duty diesel vehicles in the Australian market. This is in line with international (European) practice. The Australian Biofuels Taskforce report noted that warranty acceptance is a key factor in growing the biodiesel industry domestically (19).

The Federal Chamber of Automotive Industries (FCAI) has provided the following statement:

FCAI is the peak industry body that represents the majority of Australia's manufacturers and importers of passenger and light commercial vehicles, and motorcycles. Australia is one of the most competitive automotive markets in the world with more than 50 brands, 350 models from 20 source economies. The FCAI acknowledges the important role properly refined biofuels (i.e. those conforming to the national fuel standards) can play in the transport fuels equation. The position regarding biodiesel is summarized as follows:

- FAME, including vegetable derived esters (VDE), is generally acceptable when blended with conventional diesel fuel up to 5 vol%;
- The FAME on which the biodiesel is based must comply with either EN 14214 or ASTM D6751 standards;
- The resultant B5 blend must conform to the national Diesel Standard, which is based on EN590. This is consistent with the WWFC;
- FCAI does not generally support use of B100;
- FCAI members will not warrant damage caused by using biodiesel blends greater than B5, unless such use is sanctioned by a manufacturer. Adoption

of WWFC recommendations is particularly relevant in Australia where diesel engine technology comes entirely from overseas sources;

- FCAI recommends that national standards for FAMEs and biodiesel blends, based on the EN and/or ASTM standards, be developed concurrently with a study on the impact of biodiesel on vehicles. This must include the issue of oxidation stability, regardless of the percentage of biodiesel contained in the blend; and
- There needs to be a transparent process to allow consumers to make an informed choice on whether their vehicles can use biodiesel.

The Australian specification of February 2006 has closely followed the European EN 14214 specification.

10.3.2. Japan

Japan has had biodiesel used in dedicated fleets in partnerships, such as between waste removal trucks and processors of used oils. For more general use the Ministry of Economics, Trade and Industry (METI) has taken an approach to support use in blends of B5, but coupled with controls of producers and sellers, backed by government inspection. The Japanese standard was developed based on possible problems and limiting aspects that would lead to problems as given in Table 13 below.

Table 13: Biodiesel Possible Problems addressed by Specifications in Japan

Possible problems	Parameters to control
Damage to fuel lines Metal corrosion Elastomer swell	Methanol Acid number Water Ester content Oxidation stability
Blockages and deposits	Water Ester content Oxidation stability Poly unsaturated fatty acids Mon, diglycerides Triglycerides Glycerol Solids (foreign material)
Low temperature flow problems	CFPP
Worse emissions	Triglycerides Metals
Degradation of exhaust after treatment devices	Metals Phosphorous

They specifically found the oxidation stability of the blend, with biodiesel that complies with EN 14214 depends on the petroleum diesel and required this to be tested on the blend. They therefore introduced a voluntary standard for biodiesel, JASO/JIS based on EN 14214. They added that oxidation stability and CFPP of the pure biodiesel must be

specifically addressed between the biodiesel producer, the petroleum diesel producer and any marketers involved.

They focused rather on tests of the B5 blends (12). They added tests for the B5, over and above the conventional diesel specification; for FAME (5 wt% max), triglycerides (0.01 mass% max), methanol (0.01 wt% max), TAN (0,13 mg KOH/g), individual organic acids (30 ppm max, as formic, acetic and propionic acid) and oxidation stability increase (modified ASTM D 2274) in which the acid number must not change by more than 0.12 mg KOH/g. Tests against this limit for the blend showed that some EN14214 B5 blends do not comply. The main problem was the blend oxidation stability, the increase in acid number. This can probably be addressed by suitable oxidation inhibitors (13), but more work is required.

10.3.3. Malaysia

Malaysia is the world's largest producer of palm oil, using 4 million ha (60% of their agricultural land) to produce about 15 billion liters/year of crude palm oil (CPO), or 50% of world production. They currently export 90% of this and this earned about US\$8 billion in 2005 (price ca. US\$400/ton).

Palm oil contains lower levels of olefins ($IV < 80$) and high concentrations of the C16:0 fatty acid, palmitic acid and C18 fatty acids. This makes the oil relatively stable but it has inferior cold flow properties to coconut or palm kernel oil, that are mainly C12 and C14, or rapeseed that is mainly C18 acids based, but is more unsaturated.

For the local biodiesel market, they have focused on low-level blends (B5) of a processed palm oil, Palm Olein and PME. Upon distillation of CPO, an olefin rich fraction called Palm Olein and a saturate rich fraction known as Palm Stearin can be obtained. The latter has a high melting point and would not normally be used for biodiesel. Palm Olein has good flow properties and a modest level of olefins (iodine number < 110). For export markets they either transesterify the palm oil, or export the palm oil and it is esterified by the user nations (62). The MPOB conducted extensive tests from 1982 to 1995, using different formulations and comparing it to petroleum diesel. They then declared it as a satisfactory diesel substitute. They have developed technology to lower the pour point from typical $+15^{\circ}\text{C}$ to -21°C . The viscosity of both is similar at $4.5 \text{ mm}^2/\text{s}$ at 40°C . The product complies to the EN 14214 and ASTM D 6751 specifications, with a cetane number of 62, iodine value of 52. They are targeting the EU-25 market of about 11 billion liters pa in 2010 (at 5.75% biodiesel). Together with Indonesia they target to limit the use about 40% of current palm oil (6 million tons/year) for biodiesel, so as to protect the food market. This is controlled by biodiesel manufacturing licenses. As regards increased production, there is an issue of whether natural forests are being cut down, but the Malaysian government has stated that they rather use conversions of other crops (2 million ha available) and better technologies and management to increase yields. With the currently announced biodiesel capacity, Malaysia would be able to support B5 in transport fuels and have a small quantity of

finished biodiesel available for export in addition to the export of palm oil for processing in export markets.

10.3.4. Philippines

The Philippines is using the biodiesel to revive their coconut industry, as use of the oil had been declining (16). This oil has fatty acids of much lower molecular weight than other vegetable oils with a high proportion of <C14:0 fatty acids, in fact mainly C12. Although these are saturated (hence of high oxidative stability) the lower molecular weights improves the cold flow properties relative to palm oil or tallow.

The NREL of the U.S. assisted the Philippines by running a CME biodiesel program (63). This found that stability was similar to mineral diesel in blends and as B100. The flash point of CME is typically about 105°C, so changes are required in certain national specifications and the EU to allow its use. This is far higher than the flashpoint specification of mineral diesels, so the Brazilian approach of focusing on the blend would enable use of CME. The viscosity is about 2.5 mm²/s at 40°C, so is also lower than the EN 14214 requirement, but in excess of the limit for mineral diesel. There does not appear to be a reason to have a differing limit for biodiesel, apart from limiting the feedstock choice. The lighter CME also has a low 95% distillation of 327°C. The study found that 5 vol% CME blends complied with the standard Philippines and U.S. diesel (D2) specifications. It also did not mix with water, particularly useful as the Philippines sometimes use salt water to push diesel through pipelines.

The CME falls under the FAME category, and is accepted by the WWFC. A Philippine national standard (PNS 2020:2003) has been developed and this enables blends of 1 vol% to 5 vol% with mineral diesel to comply with the national diesel standard. The Philippines currently mainly use B1 blends. They have found, based on use in 1,479 government vehicles, that this gives a 17% average improved fuel economy (varying from 3%–5%). The improvement is higher for older vehicles, allegedly due to the cleaning effect. The polar biodiesel acts has good solvency and acts as a detergent additive in the diesel and cleans injector nozzles and removes combustion chamber deposits. It has further been shown to results in about a 50% smoke reduction, and this is supported by typical 70% reductions in PM (62). They surprisingly even found a 20% NO_x reduction. A contributing factor is the CN of 70, but this has a minimal effect in a 1% blend. Individual engine dynamometer testing has shown a 1%–3% power increase.

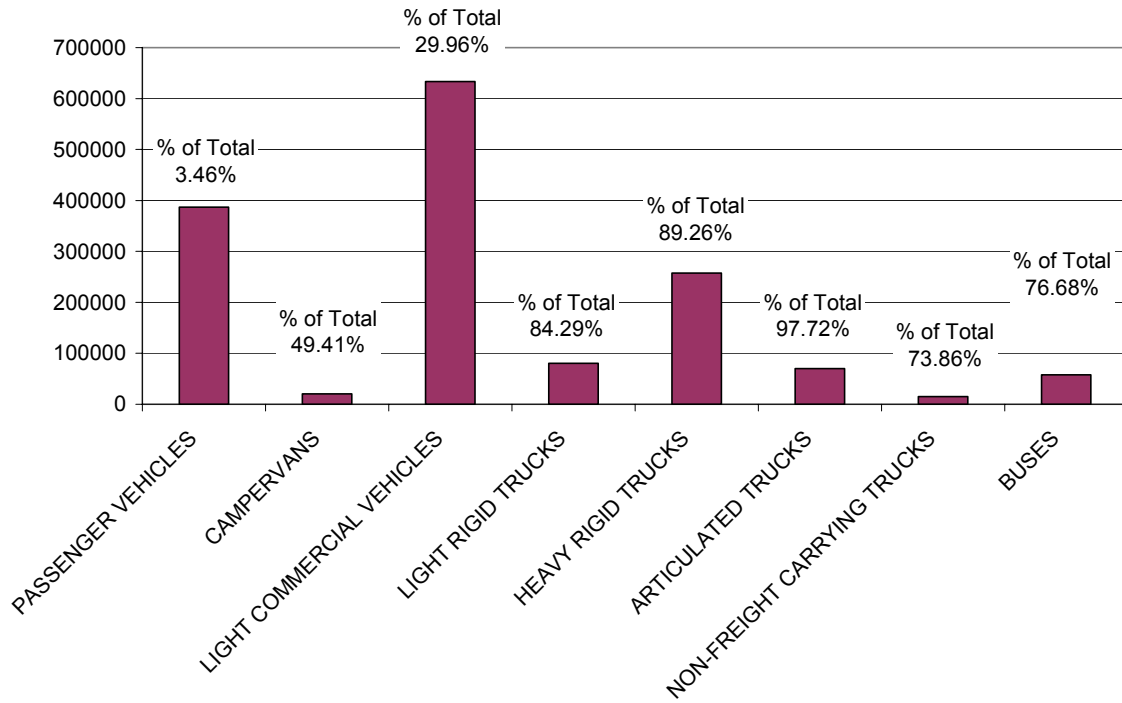
10.4. Vehicle Fleet Information by Economy

10.4.1. Australia

The average annual growth in 2006 was 2.9%. The average age of all vehicles registered in Australia at March 31, 2006, was 10.1 years. Vehicles manufactured before 1991 (more than 16 years old) comprised 22.9% of the total Australian fleet. Campervans were the oldest vehicles registered with an average age of 18.9 years. Light commercial vehicles made up 41.6% of vehicles registered as using diesel fuel,

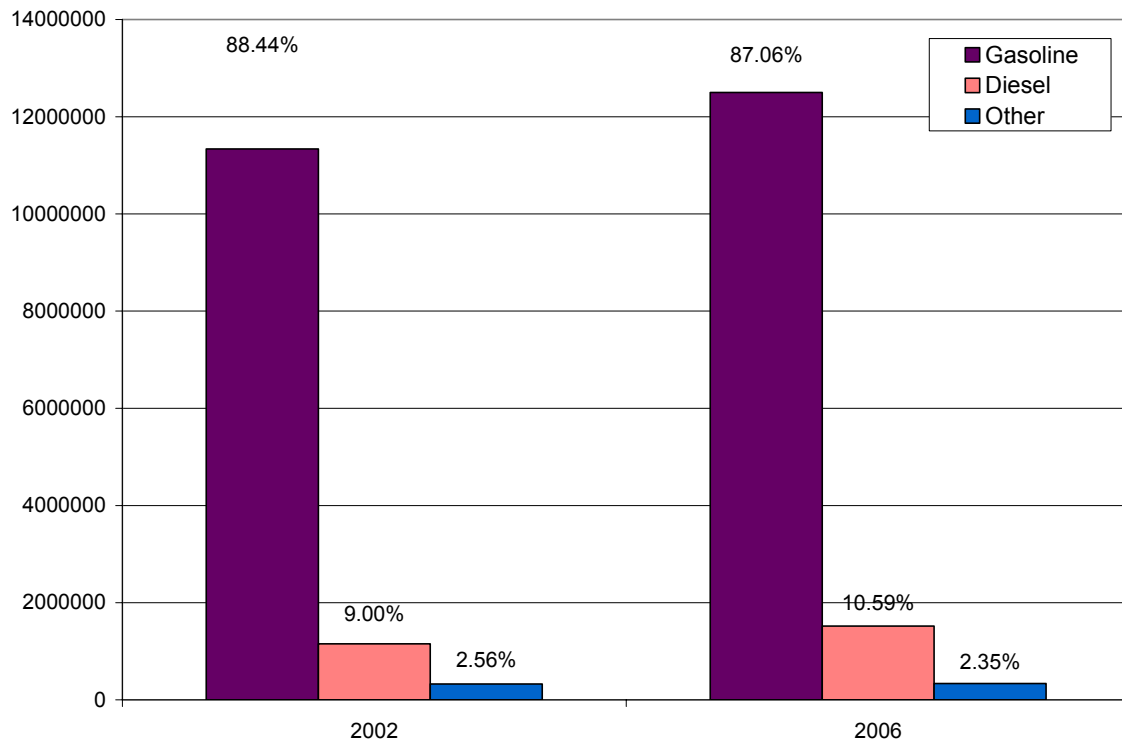
while rigid and articulated trucks accounted for 22.2% and 4.6% respectively. Passenger vehicles registered as using diesel fuel represented 25.4% of all diesel vehicles in 2006.

Figure 10: 2006 Diesel Fleet Composition by Type in Australia



Source: Australian Bureau of Statistics

Figure 11: Ratio of Vehicle Fleet by Fuel Type in Australia

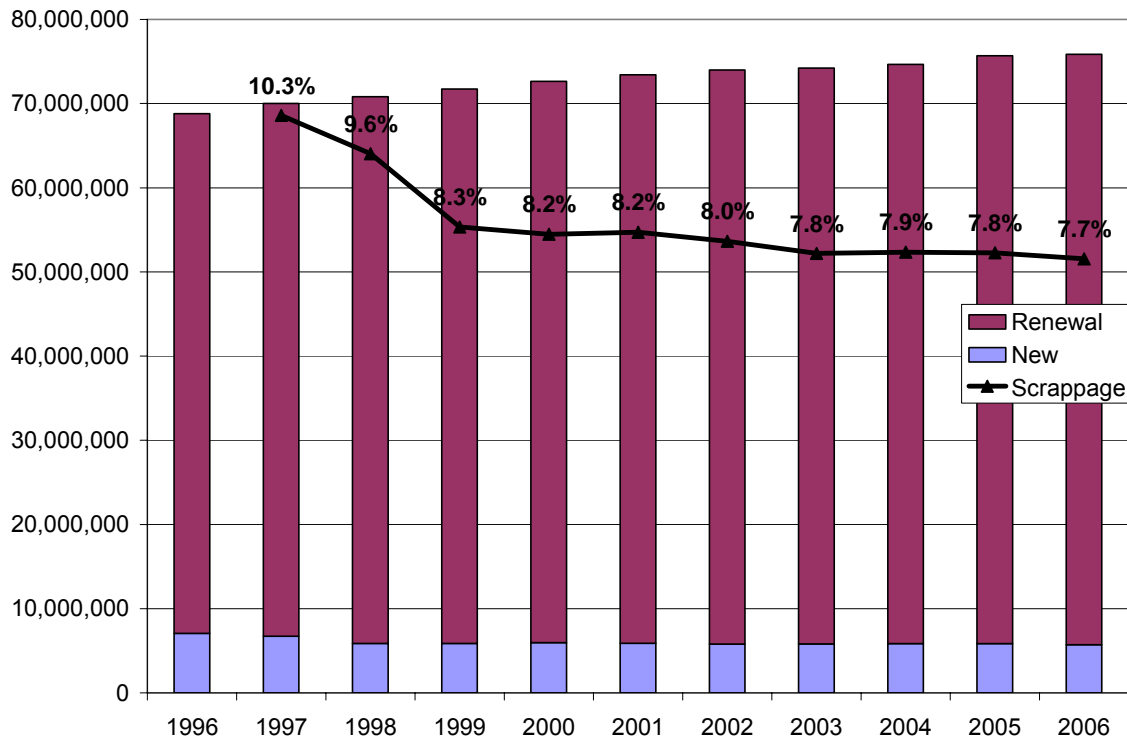


Source: Australian Bureau of Statistics

10.4.2. Japan

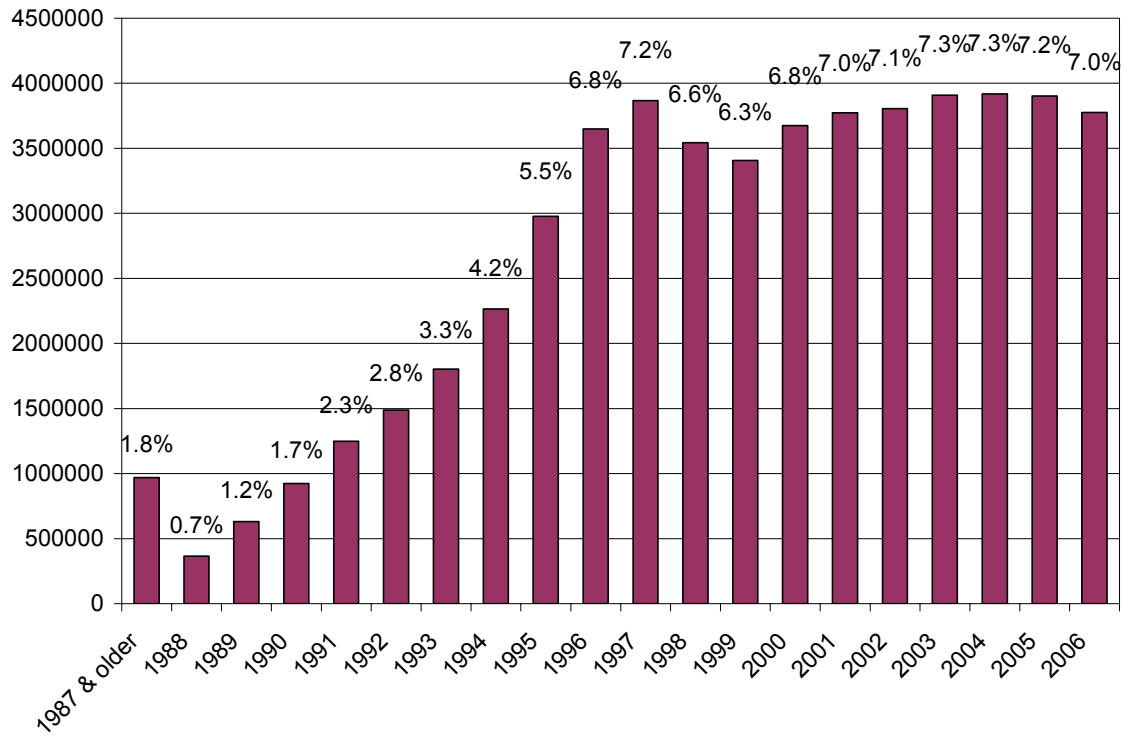
Japan has one of the youngest fleets in the region. A high scrappage rate, helped by a robust inspection and maintenance program makes adoption of non-conventional fuels to the on-road fleet less problematic. Issues can be identified in advance and sufficient warning is provided to operators of vehicles expected to experience operational problems.

Figure 12: Vehicle Population and Scrappage Rate in Japan



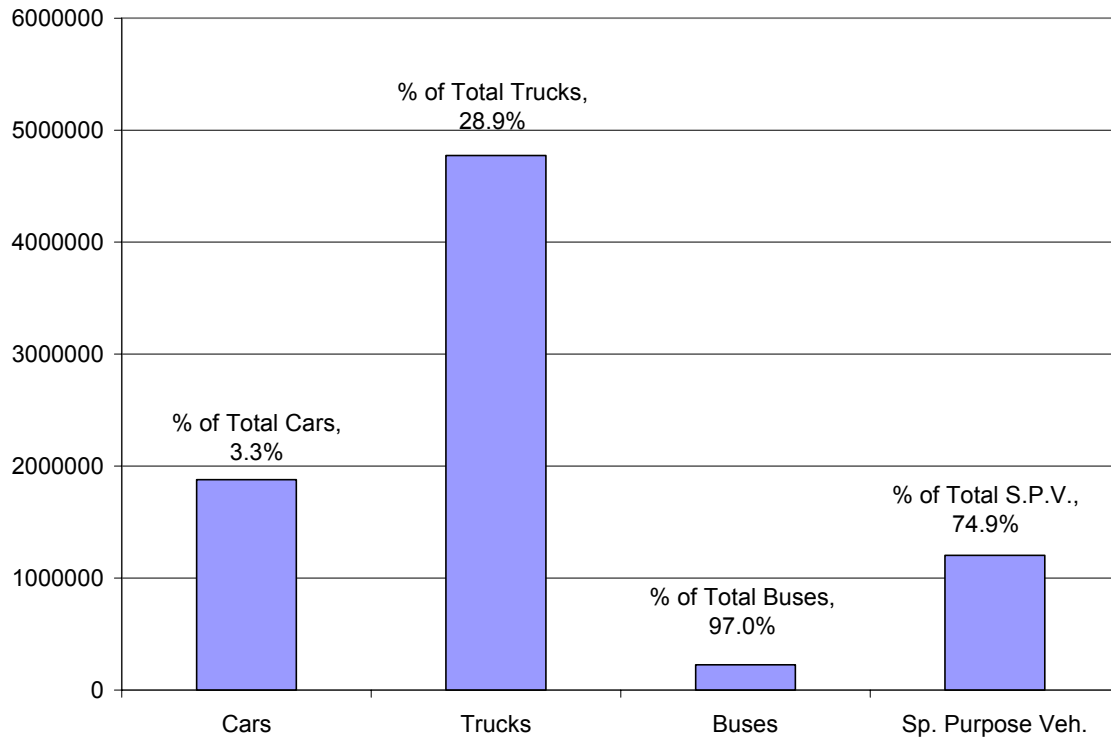
Source: Japan Automobile Manufacturers Association, Inc.

Figure 13: In-Use Diesel Vehicles, 2006 Fleet Distribution in Japan



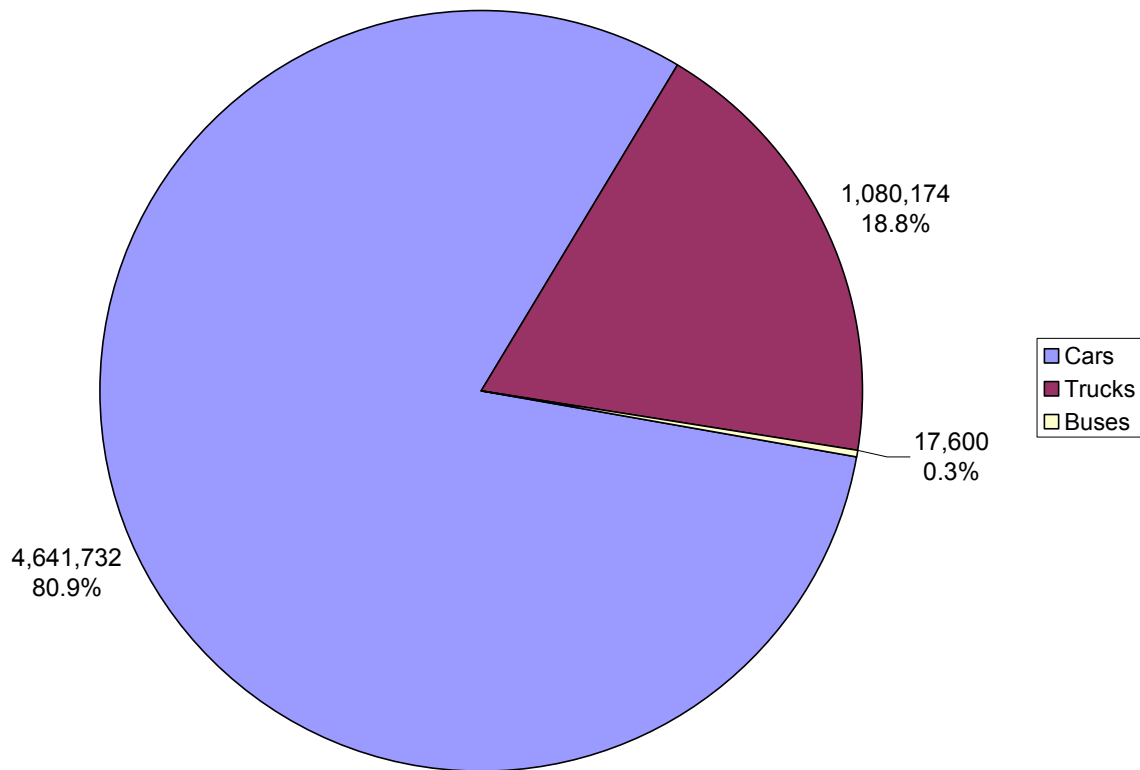
Source: Japan Automobile Manufacturers Association, Inc.

Figure 14: In-Use Diesel Vehicles by Type in Japan, 2006



Source: Japan Automobile Manufacturers Association, Inc.

Figure 15: Fleet Distribution of New Vehicles in 2006

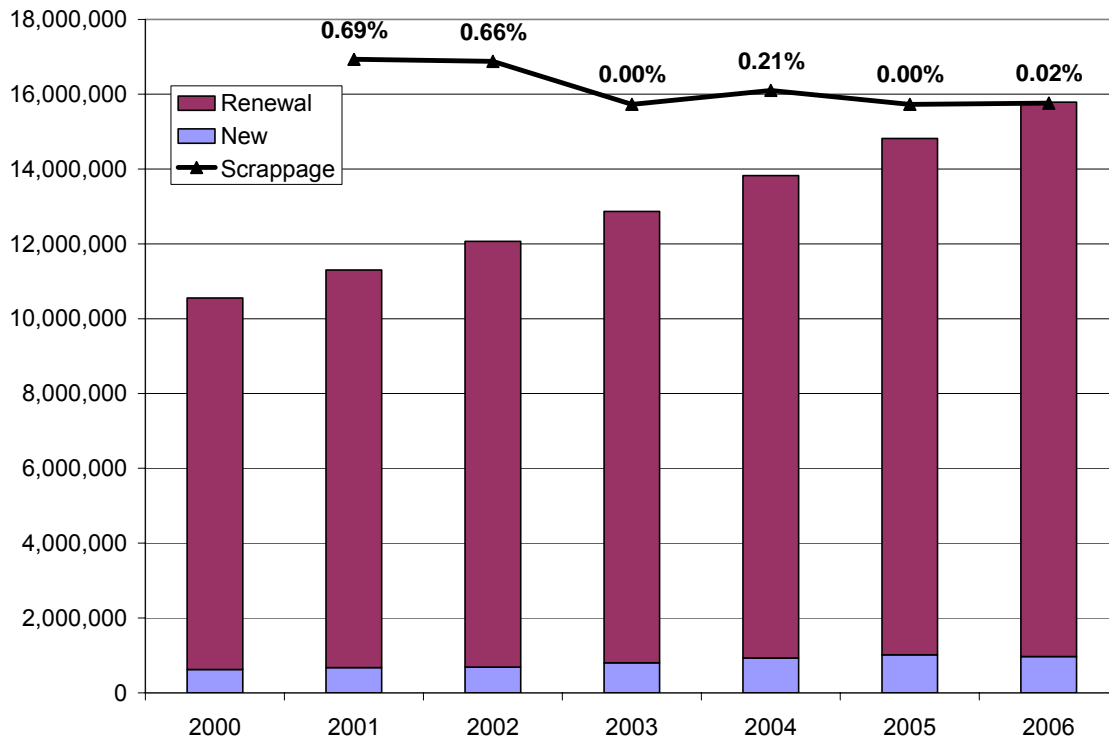


Source: Japan Automobile Manufacturers Association, Inc.

10.4.3. Malaysia

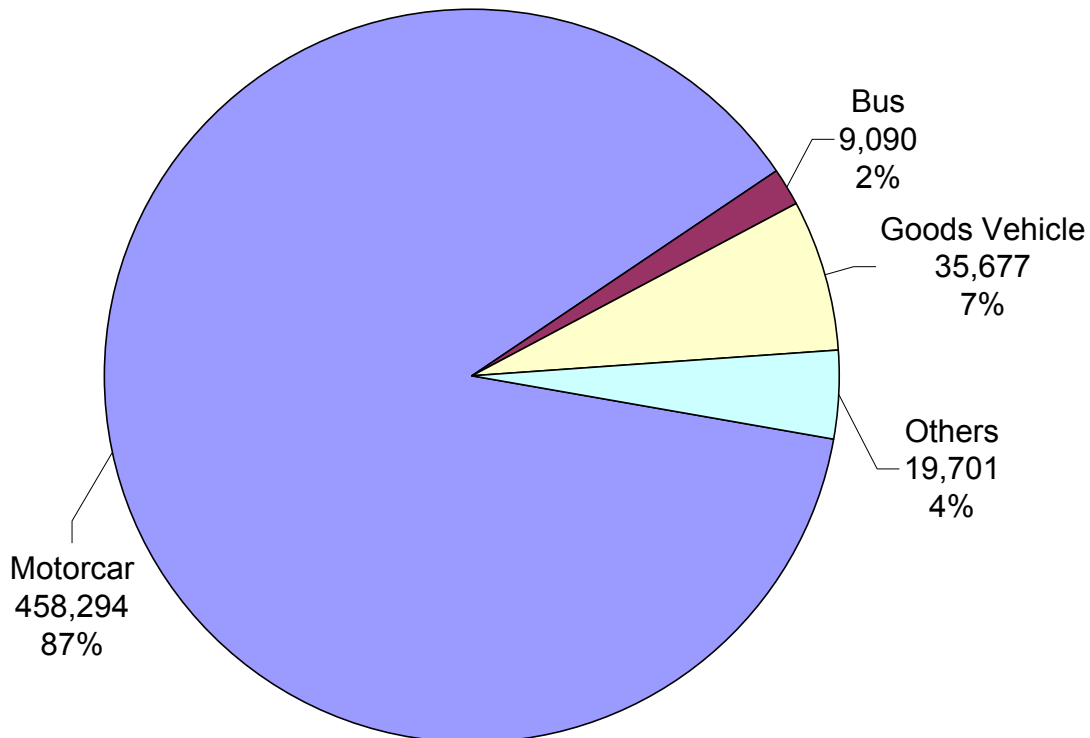
Malaysia has a registered fleet of 15.7 million vehicles. The diesel vehicle fleet is expected to constitute only 3% of the total, with the largest share being that of two wheelers. The extremely low scrappage rate makes this a constantly aging fleet. Older vehicles would tend to have more material compatibility issues with biodiesel.

Figure 16: Vehicle Population and Scrappage Rate in Malaysia



Source: Road Transport Department

Figure 17: Fleet Distribution of Newly Registered Vehicles in Malaysia, 2006

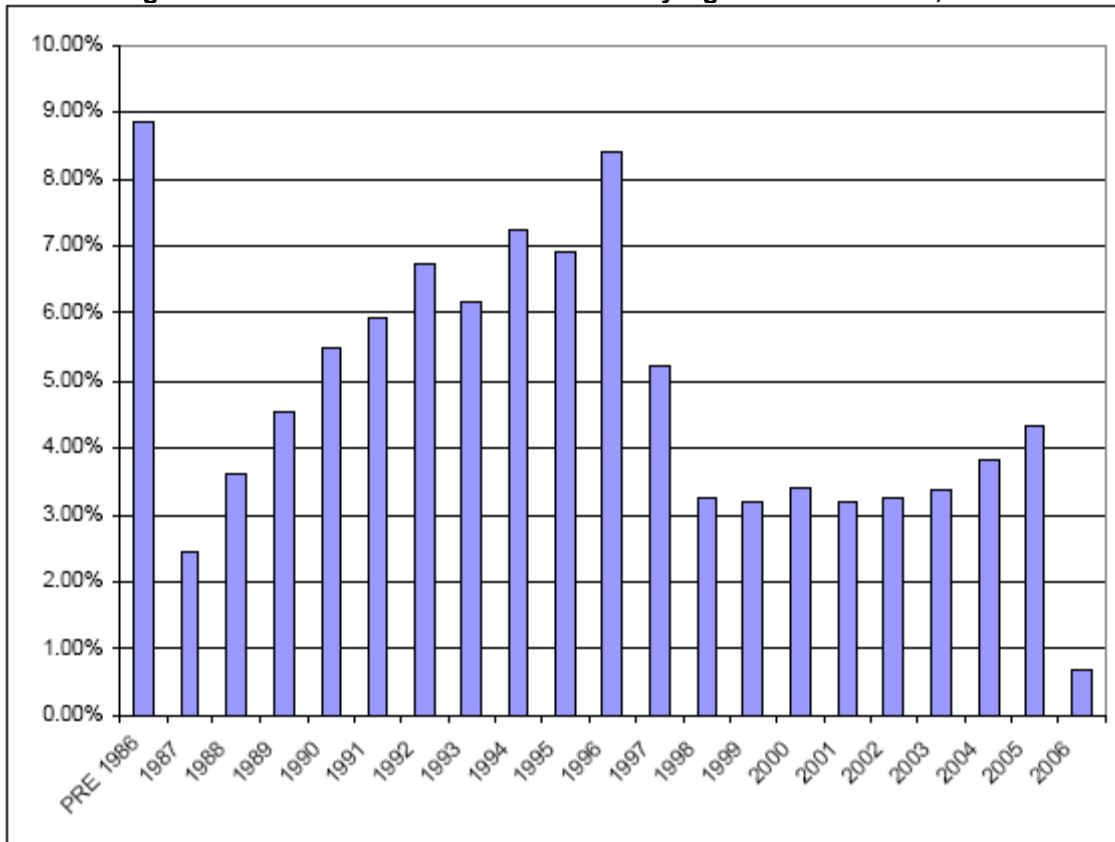


Source: Road Transport Department

10.4.4. New Zealand

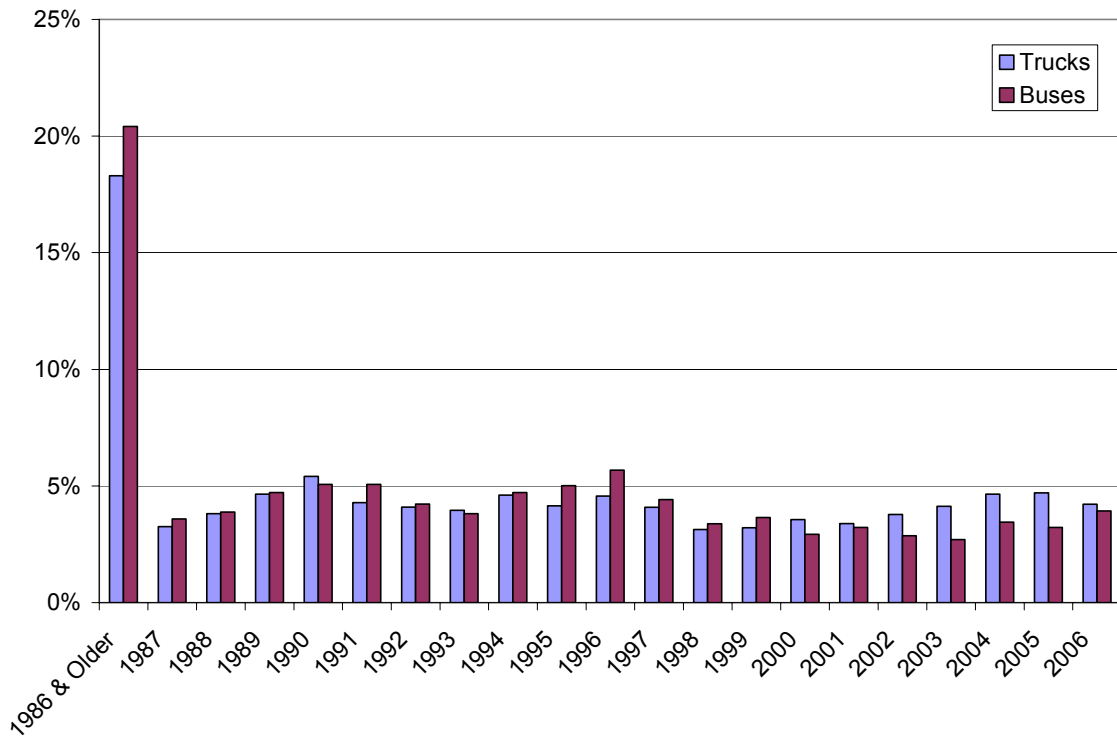
New Zealand saw a large growth in the 1990s for diesel vehicles. This has attributed to the uncommon diesel vehicle distribution with high concentration of mid aged diesel vehicles. Cars and trucks form the bulk of the diesel fleet with scrappage rates of 4.8% and 3.7% respectively. Since, most of the vehicles on-road in New Zealand are expected to be European; introduction of B5 in the fleet is not expected to pose any technical challenges to the vehicles.

Figure 18: Distribution of Diesel Vehicles by Age in New Zealand, 2006



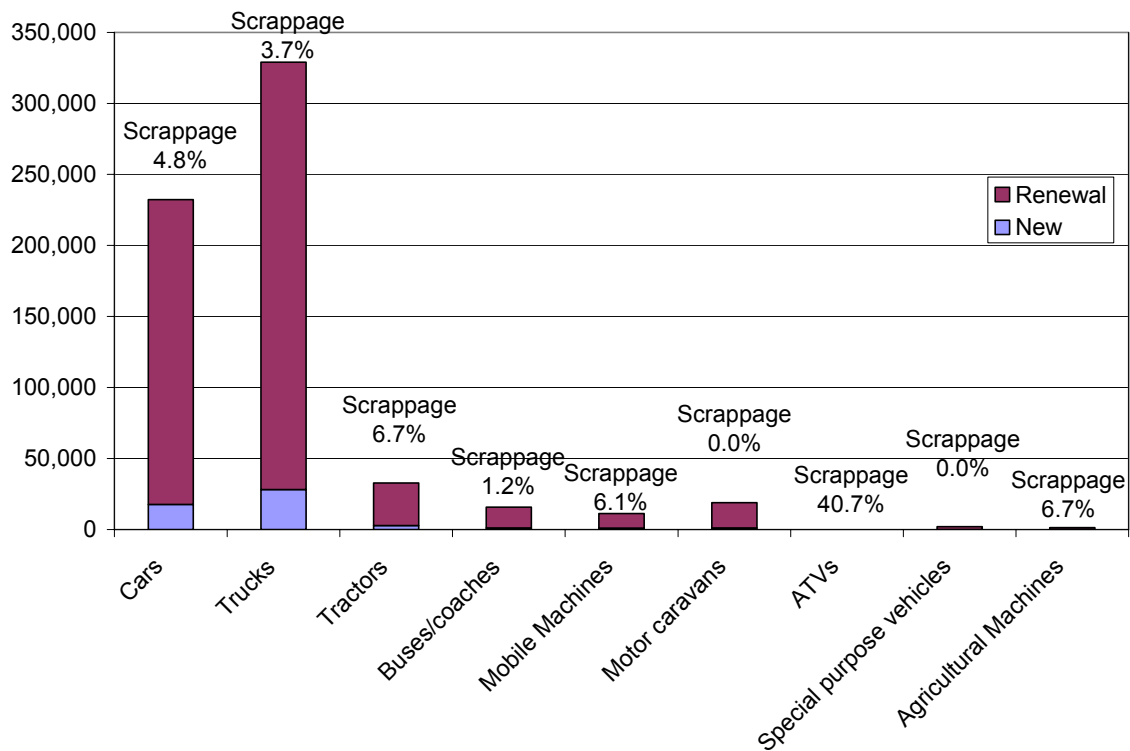
Source: Land Transport New Zealand

Figure 19: Distribution of Trucks and Buses by Age for New Zealand, 2006



Source: Land Transport New Zealand

Figure 20: Diesel Vehicle Distribution by Type and Scrappage Rate for New Zealand, 2006

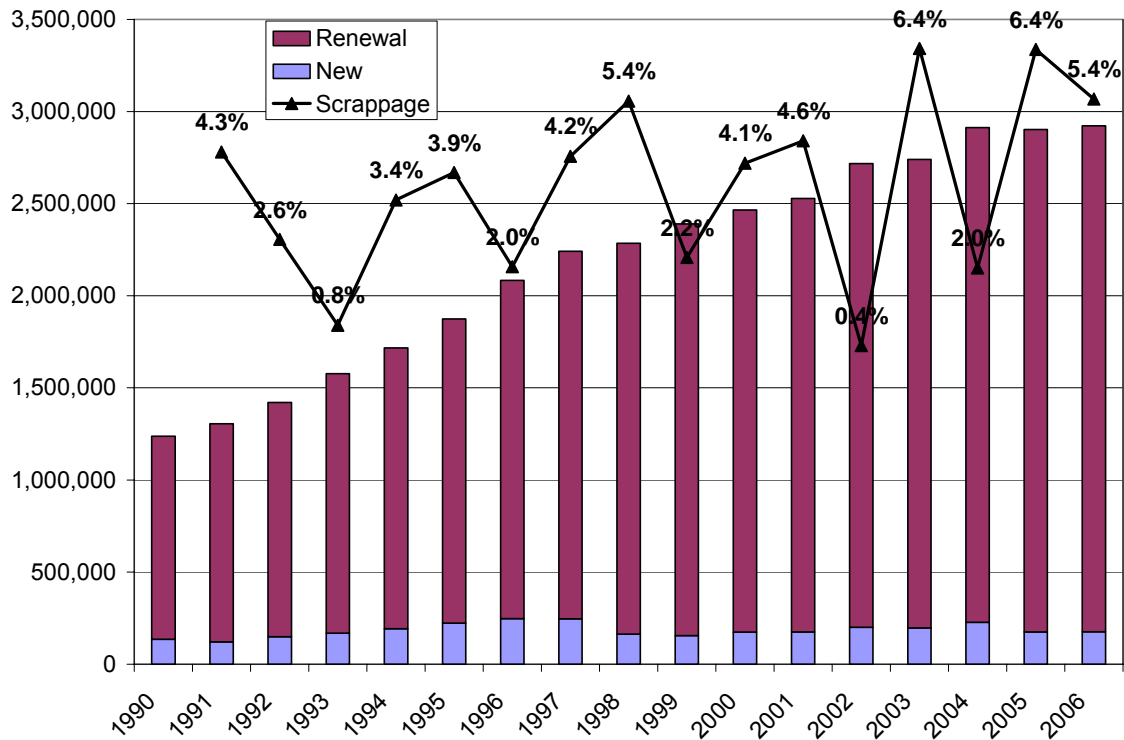


Source: Land Transport New Zealand

10.4.5. The Philippines

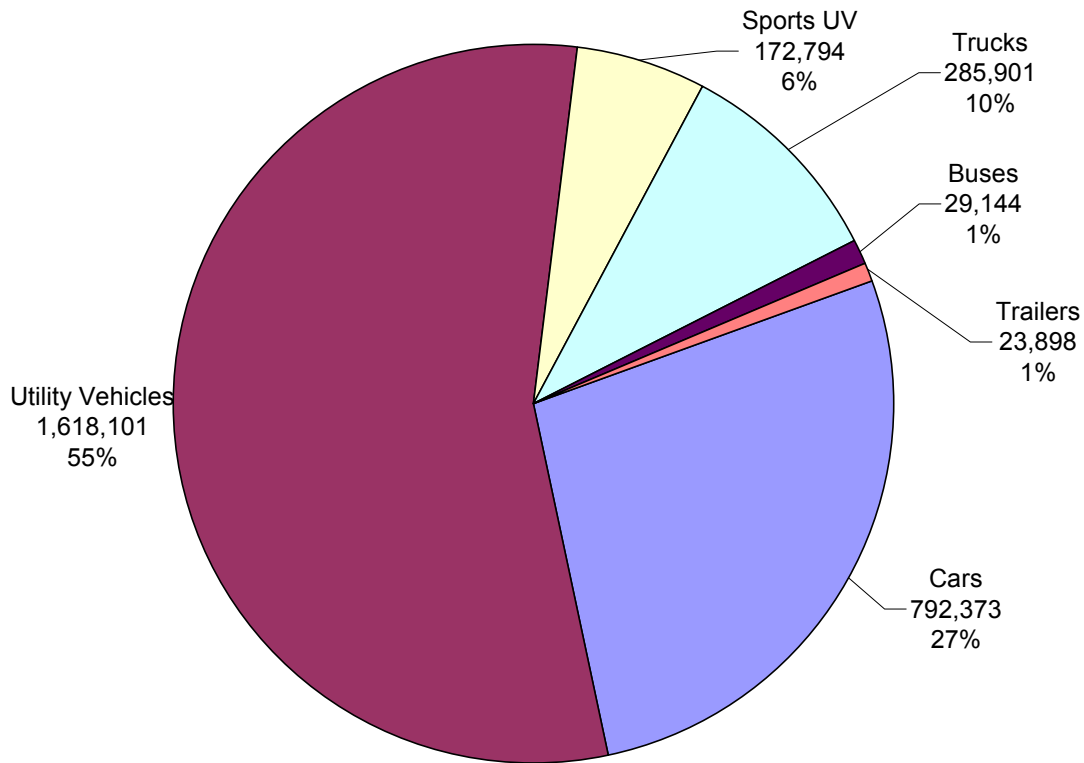
With a relatively small vehicle population as compared to similar economies, The Philippines has maintained a vehicle scrappage rate of approximately 5%. The primary users of biodiesel being buses, trucks and trailers constitute about 12% of the vehicle population (excluding two wheelers).

Figure 21: Vehicle Population and Scrapage Rate for The Philippines



Source: Department of Transportation Office, The Philippines

Figure 22: Fleet Distribution of Newly Registered Vehicles in The Philippines, 2006

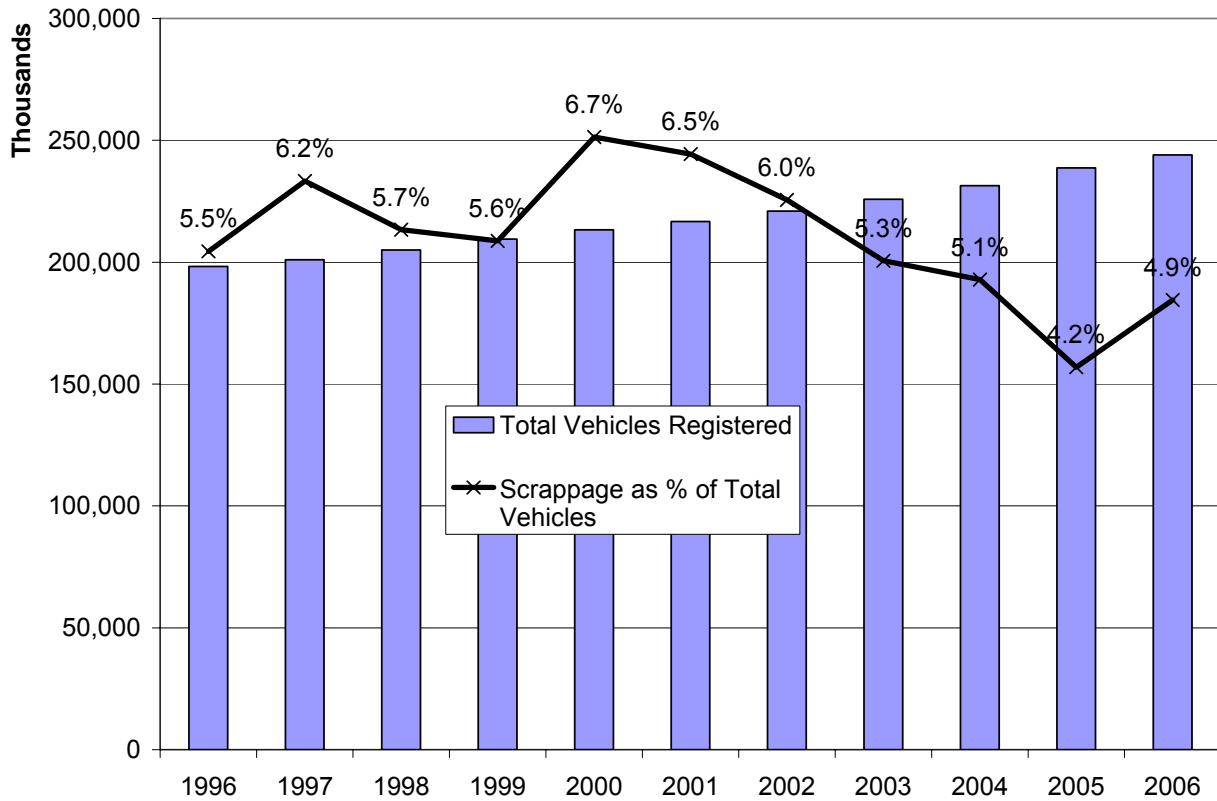


Source: Department of Transportation Office, The Philippines

10.4.6. U.S.

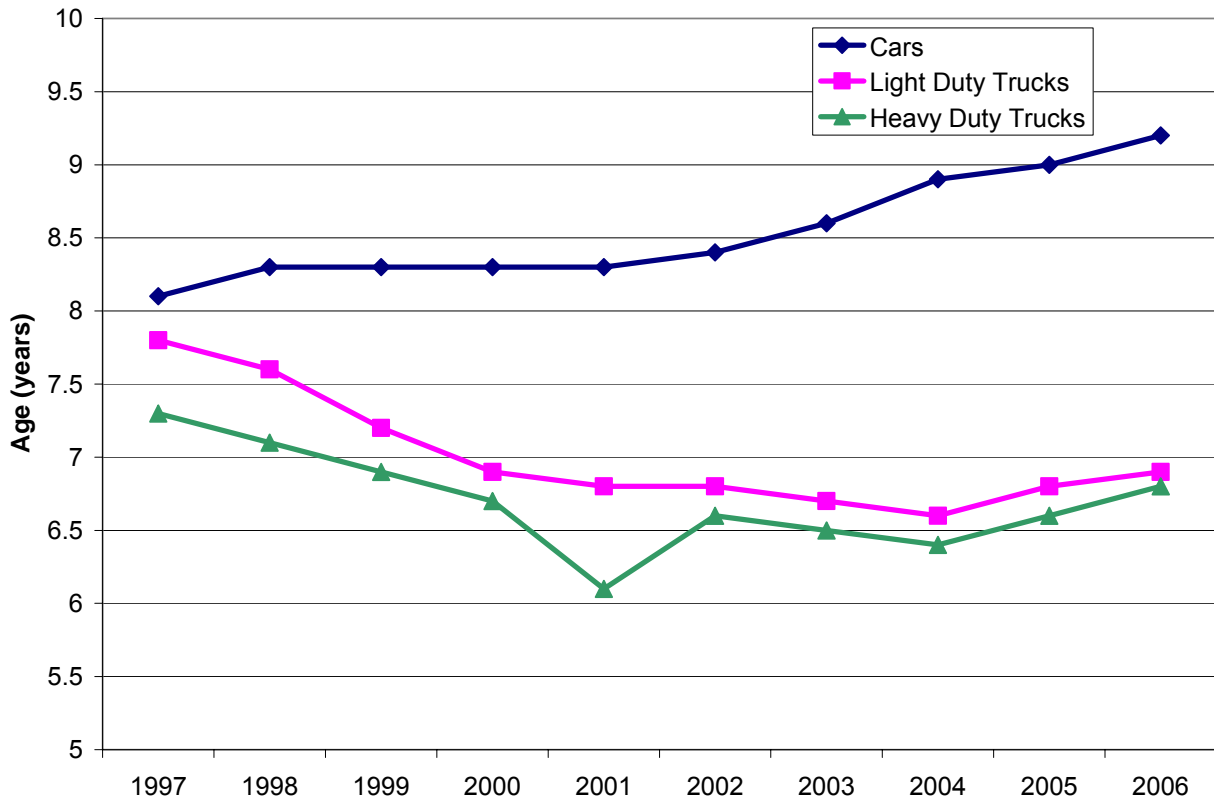
The U.S. has more than 247 million vehicles registered as of Oct, 2006. The light duty vehicles have a growth rate of a little over 2% per year, with a median age of 8 years in 2006. The scrappage rate for passenger vehicles has seen a decline over the years, at 4.9% in 2006. The median age of vehicles also increased across all major vehicle categories. The 9.2 year median age of cars continues a five-year trend. For all trucks, the median age increased to 6.9 years in 2006. Light truck median age in 2006 increased to 6.8 years. In 2005, the diesel light duty vehicles sales accounted for just above 3.5% of all light duty vehicles.

Figure 23: Light Duty Vehicle Population and Scrappage Rate for U.S.



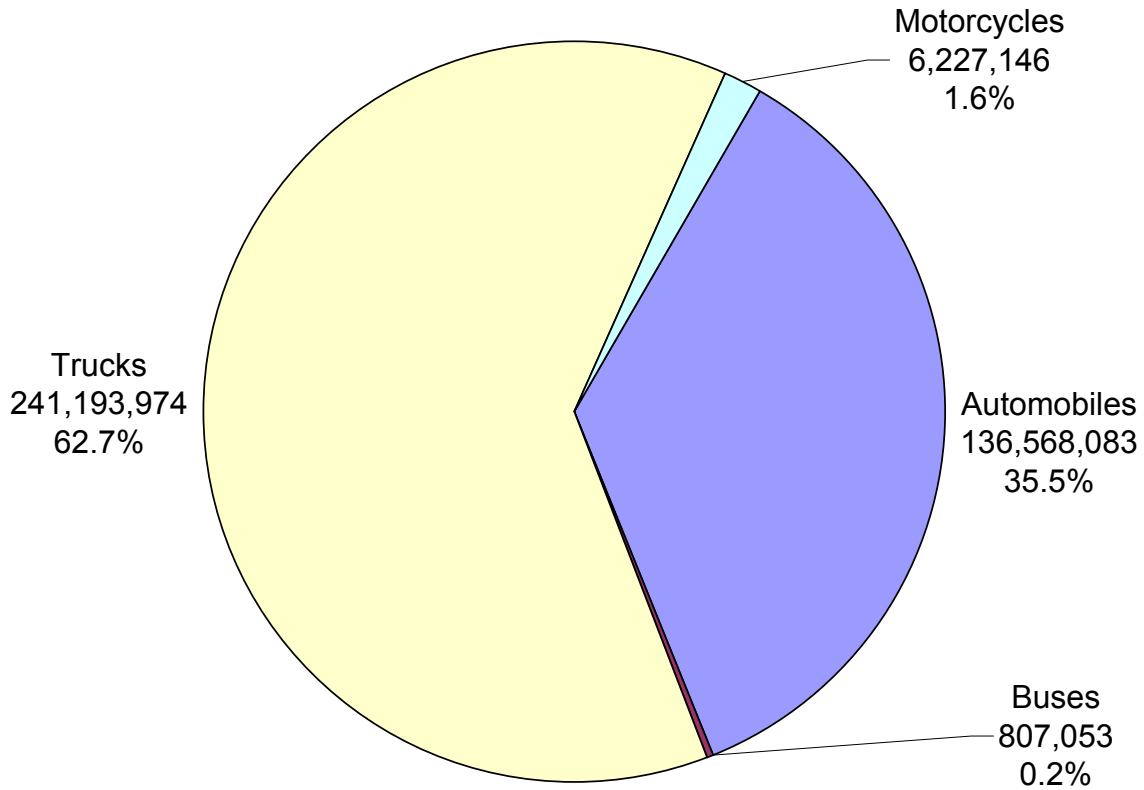
Source: National Automobile Dealers Association, May 2007

Figure 24: Median Age of Vehicles in the U.S.



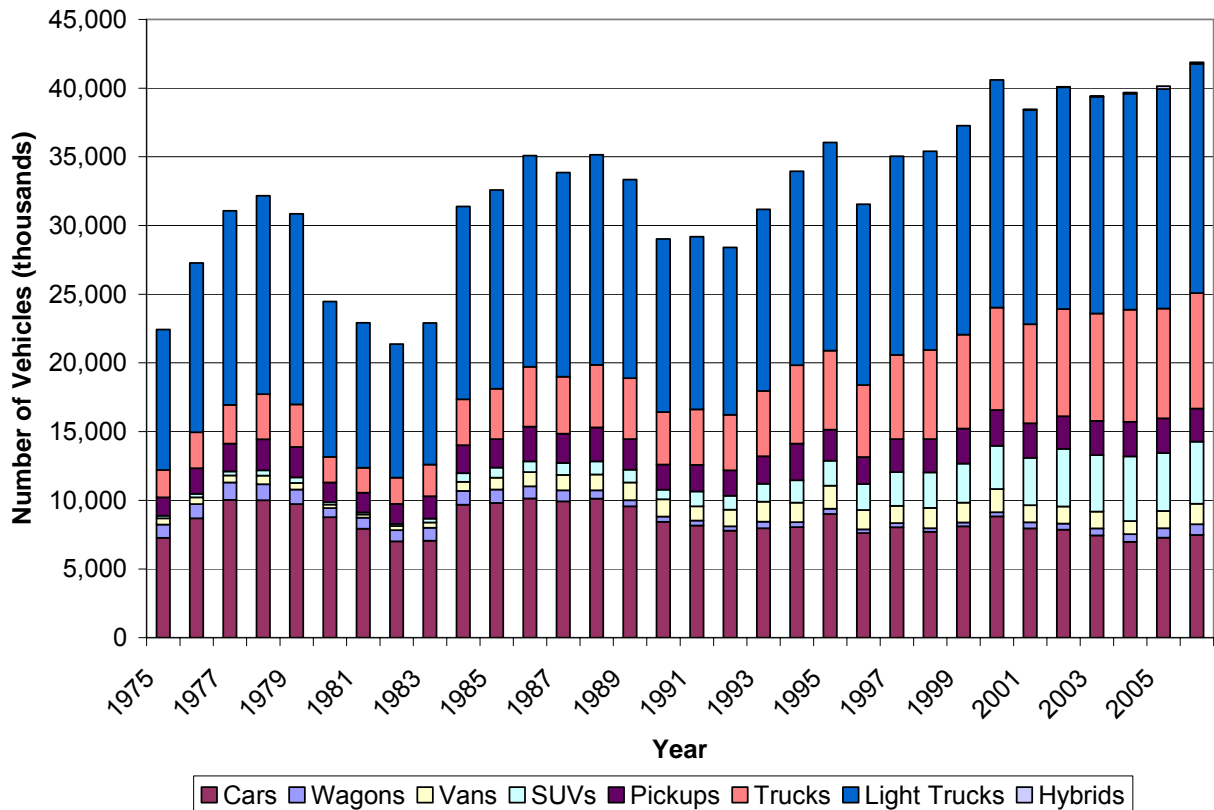
Source: R. L. Polk & Co.

Figure 25: Fleet Distribution of Registered Vehicles in U.S., Oct, 2006



Source: U.S. Department of Transportation - Federal Highway Administration

Figure 26: New Vehicle Registration Trend in the U.S.



Source: U.S. Department of Transportation - Federal Highway Administration

10.5. Quality Control Aspects⁽²³⁾

As crude oil prices went up again from early 1999 onwards, a number of new investment projects for biodiesel production were triggered and new producers and importers tried to market their methyl-ester fuels. In practice, biodiesel of substandard quality was in circulation thus creating continuous mobility problems for customers and damaging the carefully established quality image of biodiesel.

The risk for the established good image of the high quality fuel biodiesel was evident, as customers would associate this fuel with unreliability and expensive failures. Therefore the objective was set to establish a strict quality control system in order to assure fuel quality.

In order to defend the established good image of high-quality and standardized biodiesel, a registered European-wide QUALITY SEAL along well-defined criteria and

²³ This section shares extracts from the U.S. NBB BQ-900 and the European Quality Seal biodiesel quality assurance programs. A specification without proper quality control and assurance programs will be ineffective.

procedures was created (64). This quality seal is promoted as a visual signal of top quality towards the end-user at the pump, on bills and on any promotional literature; if the standard is not met, then the producer, the distributor or the selling pump is informed and may be listed as an insecure supplier or may even have to pay a penalty fee.

When methyl ester is sold at the fuel station and declared as “Biodiesel” in Europe the “Biodiesel Standard EN 14214” applies.

The ASTM is the recognized standard-setting body for fuels and additives in the United States. ASTM has adopted a specification for B100 (ASTM D6751) that is to be used in blends with diesel fuel, which meets its respective specification (ASTM D975). Typically where biodiesel is advertised (it is labeled), the advertising states that the biodiesel meets the appropriate specification, rather than commenting about the quality of the blend. In California, any diesel with greater than 5% biodiesel is seen as a development fuel so must be sold labeled as such, with the customer needing to read and sign certain conditions of use. Therefore quality is controlled generally through the quality of the individual biodiesel and diesel labeling on the blends. Biodiesel has potential problems as well. Unconverted or partly converted glycerin can result in very poor cold flow properties; in-cylinder and injector deposits; and clogged fuel filters. Methanol can degrade some plastics and elastomers, and remnant caustic catalyst in the fuel can cause excessive injector, fuel pump, piston and ring wear. All of these impurities are limited by the current ASTM D6751 specification for biodiesel. Also, because biodiesel is susceptible to oxidative and biological instabilities, a stability requirement is included in D6751.

The U.S. based National Biodiesel Board (NBB) supported the ASTM specification development. When biodiesel that meets its specification is properly blended into diesel fuel that meets its specification, and is handled according to proper fuel management techniques, the resulting fuel is a high quality, premium diesel fuel that has been shown to perform well in virtually any unmodified diesel engine. However, use of any fuel that does not meet its quality specifications could cause performance problems or equipment damage, and this includes biodiesel. The NBB believes strongly that rigorous adherence to D6751 is important in order to protect consumers from unknowingly purchasing substandard fuel, in order to maintain the integrity of the nation’s fuel supply, and in order to protect the reputation of biodiesel as a high quality, high performance fuel. Sale of off-specification fuel is usually a violation of federal and state law. Several federal and state government agencies are responsible for the regulation and enforcement of fuel quality in the United States. The Internal Revenue Service (IRS) has an active enforcement division for fuel compliance, which includes approximately 130 Fuel Compliance officers nationwide, who routinely enforce fuel issues related to gasoline and diesel fuel. The IRS currently coordinates with the American Petroleum Institute on issues related to fuel compliance. ASTM D6751 compliance is required of biodiesel gallons on which Biodiesel Tax Credit is claimed. The blender claiming the credit is required to obtain from the biodiesel producer a certificate stating, under

penalty of perjury, that the biodiesel or agri-biodiesel is properly registered with the EPA and meets the requirements of ASTM D6751.

The NBB has set up BQ-9000 program for marketers. The BAC (Biodiesel Association of Canada) and CRFA (Canadian Renewable Fuels Association) have endorsed the BQ-9000 Quality program and some OEM's are starting to include this requirement in their warranties.

10.5.1. BQ 9000 Quality Program

The NBB backs the development of BQ9000 programs to assure quality is maintained from production to consumption. The driving force in both the product specifications and the quality programs is to develop confidence in biodiesel amongst the petroleum producers and distributors as well as the diesel engine OEM's.

QA and QC, based upon the ASTM or CEN specifications, is mandated and determined by process monitoring and on-site lab testing. The goals are:

- To promote the commercial success and public acceptance of biodiesel
- To help guarantee that biodiesel fuel is produced and maintained at ASTM D6751 levels
- To provide a mechanism to track biodiesel in the distribution system, identifying biodiesel which meets ASTM standards.

In this they indicate a requirement for a quality manual, quality policy and documented quality system procedure with internal and external auditing in place. This is to demonstrate an ability to provide product that meets ASTM D6751.

This includes processes for corrective action and the prevention of nonconformity.

It applies equally to producers or marketers

It provides confidence to customers that intended quality is met through demonstrated capabilities.

Program is designed to monitor the production of biodiesel to the ASTM D 6751 specification including:

- sampling,
- testing,
- retention samples, and
- shipping.

Under sampling and testing they indicate:

- ALL TANKS: density top middle and bottom as an indicator of homogeneity with limit of 0.006;
- ALL PRODUCTION LOTS: appearance, free from particulate matter, water, unreacted material. Full specification to be carried out on every lot until it is proven that product is consistently in specification (minimum of 7 consecutive

lots). At this point the manufacturer can switch to critical testing and monthly testing. If a process change occurs the manufacturer has to re-establish confidence over three consecutive lots;

- CRITICAL TESTS UNDER ASTM: Appearance, flash point, water and sediment, cloud point, acid number, free glycerol, total glycerol, sulfur and oxidative stability;
- EVERY MONTH: Na, K, Ca and Mg; and
- EVERY SIX MONTHS: Full panel of tests.

10.5.2. BQ-9000 Accredited Marketer

The marketer can purchase biodiesel from an NBAC accredited producer and rely on the COA generated by the producer, or the marketer can purchase the biodiesel from a non-accredited producer and have the testing performed to produce a valid COA.

Product received from an NBAC accredited producer may bypass the ASTM D 6751 tests required to generate an additional COA upon receipt and be off-loaded directly into a loaded directly into a distribution tank. The producer generated COA applies to this product.

A representative sample shall be taken, as the truck is off-loaded into the distribution tank. The sample shall be visually inspected for water, sediment, and particulate matter.

Biodiesel according to EN 14214 is produced and sold by numerous manufacturers in Germany and neighboring EU economies. However, practices for monitoring the quality of these products vary widely. The Bio-Diesel Quality Management Work Group (AGQM), a registered association, was founded in 1998 to raise users' confidence in biodiesel. This association comprises a voluntary group of biodiesel manufacturers implementing consistent quality assurance as a leading corporate policy and assisting other market participants in supplying high-quality biodiesel to users. The AGQM's network now includes about 1400 public filling stations offering consumers biodiesel of an assured quality and guaranteeing that batches can be traced if they ever prove deficient (further details on the AGQM's activities and members are available at www.agqm-biodiesel.de). To monitor and improve quality, the AGQM is continuously advancing their quality management system. This includes spot checks of products ranging from the manufacturer's outlet through to intermediate warehouses and to filling stations, annual audits, provision of information and educational courses. And, in particular, ensuring adherence to limiting values agreed beyond those specified by EN 14214. For example, the biodiesel manufacturers organized under AGQM guarantee that winter consignments are delivered already 4 weeks prior to the deadlines specified in the related standard. Stricter requirements are also imposed on water content and total contamination. Biodiesel intended for public filling stations must comprise rapeseed methyl ester furnished with an oxidation stabilizer. Every batch is delivered together with a factory certificate or analysis indicating the batch's test values.

Biodiesel in AGQM quality has become synonymous for successful quality assurance of biodiesel. Contracts regarding a supply of biodiesel should:

1. contain mandatory and testable specifications on product quality, and
2. specify procedures agreed by both parties in response to actual or presumed deviations from standards.

This includes clear labeling of supplied products: The designation "biodiesel" alone is not sufficient and must be accompanied by at least a reference to EN 14214. Deliveries required to comprise exclusively rapeseed methyl ester must be declared explicitly as such. Suppliers should clearly describe their internal quality assurance measures (especially as regards to batch tracing). All products intended for sales at public filling stations should be furnished with oxidation stabilizer. Assurance of high oxidation stability ex works alone does not guarantee fulfillment of specifications on transfer of the product to the final customer. Delivery quality should be borne out by an updated, concise, batch-specific factory certificate or analysis. In the case of biodiesel intended simultaneously for several suppliers, it is advisable to determine, for example, whether different flow improvers for adjusting low-temperature stability are in use in order to assess the potential for incompatibility (34).

As part of their product-related responsibilities, AGQM members ensure a continuous supply of all necessary information to users.

10.5.3. Key Issues for Controlling Biodiesel Consistency and Quality

Equipment and expertise requirements for quality control of production factors. All biodiesel production facilities should be equipped with a laboratory so that the quality of the final biodiesel product can be monitored. It is also important to monitor the quality of the feedstocks. One strategy used by many producers is to draw a sample of the oil (or alcohol) from each delivery and use that sample to produce biodiesel in the laboratory. This test can be fairly rapid (1 or 2 hours) and can indicate whether serious problems are likely in the plant.

Measuring feedstock quality can usually be limited to acid value and water content. These are not too expensive (US\$1,000 for the acid value equipment and US\$5,000 for the water measurement equipment) and can be operated by less experienced technicians.

To monitor the completeness of the reaction according to the total glycerol level specified in ASTM D6751 requires the use of a gas chromatograph and a skilled operator. Large producers will find that having this equipment on-site is necessary. Commercial laboratories are available that can analyze the samples but the cost is US\$100–US\$150 per test and the time required may be several days. Smaller producers will need to use a more robust production process involving extra methanol and probably multiple reaction steps. Then the product quality can be monitored through periodic testing by an outside laboratory.

Other possibilities for monitoring the transesterification reaction and assessing fuel quality are methods based on spectroscopy (such as near-infrared spectroscopy) or

physical properties (such as viscometry). These methods, although they are not yet ASTM methods, are usually faster and easier to use than gas chromatography. However, some of them require somewhat extensive calibration. They also cannot accurately quantify glycerol at the low-levels called for in the ASTM standard. To circumvent this, comparison to a reaction and product known to meet ASTM standards is needed.

10.6. Storage and Handling Effects

Because of high level of unsaturation, biodiesel in the presence of oxygen from air produces peroxides, acids, aldehydes, and viscosity increasing polymers. Anti-oxidant additives can control this process. Current quality standards allow for storage up to six months without need for further precautions. Additional issues with extended storage of biodiesel in the fuel tank are that it can bond with water, creating acids. Biodiesel is also a good medium for microbial growth and such growth is accelerated by the presence of water. Both problems can be addressed by measures to keep water out of the fuel tank, for example, by keeping the tank full during idle periods to prevent humid air from condensing water on the tank inner walls.

10.6.1. Storage Stability

Storage stability refers to the ability of the fuel to resist chemical changes during long-term storage.

These changes usually consist of oxidation due to contact with oxygen from the air. The fatty acid composition of the biodiesel fuel is an important factor in determining stability towards air. Generally, the more unsaturated fatty acids (C18:2, linoleic acid; C18:3 linolenic acid) are more susceptible towards oxidation. The changes can be catalyzed by the presence of certain metals (including those making up the storage container) and light. If water is present, hydrolysis can also occur. The chemical changes in the fuel associated with oxidation usually produce hydroperoxides, which can, in turn, produce short chain fatty acids and aldehydes. Under the right conditions, the hydroperoxides can also polymerize. Therefore, oxidation is usually denoted by an increase in the acid value and viscosity of the fuel. Often these changes are accompanied by a darkening of the biodiesel color from yellow to brown and the development of a "paint" smell.

When water is present, the esters can hydrolyze to long chain free fatty acids, which also cause the acid value to increase. There is currently no generally accepted method for measuring the stability of biodiesel. The techniques generally used for petroleum-based fuels, such as ASTM D2274, have been shown to be incompatible with biodiesel. Other procedures, such as the oil stability index or the Rancimat apparatus, which are widely used in the fats and oils industry, seem to be more appropriate for use with biodiesel.

However, the engine industry has no experience with these tests and acceptable values are not known. Also, the validity of accelerated testing methods has not been established or correlated to actual engine problems. If biodiesel's acid number,

viscosity, or sediment content increase to the point where they exceed biodiesel's ASTM limits, the fuel should not be used as a transportation fuel.

Additives such as BHT and TBHQ (t-butylhydroquinone) are common in the food industry and have been found to enhance the storage stability of biodiesel. Biodiesel produced from soybean oil naturally will contain some antioxidants (tocopherols, i.e., vitamin E), providing some protection against oxidation (some tocopherol is lost during refining of the oil prior to biodiesel production). Any fuel that will be stored for more than 6 months, whether it is diesel fuel or biodiesel, should be treated with an antioxidant additive.

10.6.2. Comparatively Low Toxicity to Marine Plants and Animals

From 1994 through 1996, CytoCulture conducted a series of tests in collaboration with the California Department of Fish & Game (Office of Oil Spill Prevention & Response) to document the impact of vegetable methyl esters on various native species of marsh plants and marine organisms. The studies indicated that biodiesel, while not completely harmless to the larvae of crustacea and fish, is much less toxic than petroleum fuels and crude oil.

10.6.3. Biodegradability of Biodiesel in the Aquatic Environment

A study conducted at the University of Idaho in 1995 determined that rapeseed biodiesel would biodegrade about twice as fast as petroleum diesel using a standard EPA test protocol based on carbon dioxide evolution and gas chromatography. Further, the biodiesel was shown to enhance the biodegradation rate for diesel fuel in a blend. Because biodiesel is a simple, straight carbon chain with two oxygen's at one end (mono-alkyl ester), it is more readily metabolized by bacteria that normally break down fats and oils in the environment. The petroleum diesel hydrocarbons lack oxygen, and represent a very complex mixture of hydrocarbons with multiple double bonds, and many other branched, cyclic and cross-linked chains. The more complex chemical structures of diesel hydrocarbons make them more difficult to biodegrade, and in many cases, toxic.

10.7. Standards and Specifications

According to the definition in ISO (International Standards Organization), a 'standard' can be characterized as:

- a written document, approved by a recognized body,
- available to the public,
- drawn up with the consensus of all parties concerned and to the benefit of all,
- intended for repeated or continuous application, and
- normally not mandatory (except where referred to in regulations).

To ensure that standards are generally accepted and suitable for practical application some principles have to be complied to:

- Neutrality of teamwork, access and transparency: All parties affected by the standard should be able to participate at all levels of the standardization process by sending experts to the meetings;
- Consensus: The process of standardization should result in a consensus;
- Public comment: Every draft is subject to a public comment process; and
- Coherence: Standards have to be consistent.

10.8. Diesel FIE Manufacturers Common Position Statement on FAME Fuels

**Fatty Acid Methyl Ester Fuels
As a Replacement or Extender for Diesel Fuels
Diesel Fuel Injection Equipment Manufacturers
Common Position Statement 2007**

Please note that this statement supersedes all previous joint statements

Background:

Diesel fuel injection equipment (FIE) manufacturers fully support the development of alternative sources of fuel for compression ignition engines. In Europe and in the United States of America, as well as in other countries, fuel resources such as rapeseed methyl ester (RME) and soybean methyl ester (SME), collectively known as fatty acid methyl esters (FAME), are being used as alternatives and extenders for mineral oil derived fuels. Furthermore, the EU Biofuels Directive 2003/30/EC requires member states to ensure that a minimum proportion of biofuels or other renewable fuels are placed on the market.

The FIE manufacturers are aware of issues particular to FAME fuels, and have been active in the generation of standards for these fuels. At the time of the first common position statement in 2000 there existed national standards for vegetable oil methyl esters (VOME) in Austria, Italy, Germany and France. The European FAME standard EN14214 was ratified in 2003 and supersedes these national standards.

EN14214 provides the minimum requirements for FAME quality whether used as pure FAME or as a blend component. FAME may be currently blended in quantities of up to 5% in European diesel fuel according to the EN590 specification. **In order to reduce the risk of premature failure of the fuel system, FAME must conform to EN 14214.** Increasing biodiesel production capacities in the EU have enabled legislative authorities to consider increasing the maximum biodiesel blending level from 5 to 10 percent. Activities are ongoing to standardise and validate biodiesel blends with up to 10 percent biodiesel (B10) in the EU. The FIE industry considers it as essential to maintain the fuel stability level of EU-B5 (IP \geq 20h acc. to modified EN14112) also for future B10 blends. In any case an approval of B10 requires positive validation of B10 specific issues additionally.

To date, experience in Europe has been mainly associated with the methyl esters of rapeseed oil. Whether or not the service experience with these fuels will apply/extend to all FAMEs (like those derived from soybean, tallow and used frying oil) has yet to be determined.

FIE Manufacturers Concerns:

FAMEs are derived from a wide range of base stocks, resulting in a similarly wide range of finished fuel characteristics.

Amongst the concerns of the FIE manufacturers are the following fuel characteristics:

- free methanol
- water
- free glycerine
- mono, di- and triglycerides
- free fatty acids
- total solid impurity level
- alkali/alkaline earth metals
- oxidation stability

All FAMES are less stable than mineral oil derived fuels. FAMES are readily “bio-degradable” in the event of accidental spillage or leakage, which is claimed to be a marketing advantage. On the other hand, the reduced thermal oxidative stability is of major concern to the FIE manufacturers, as the products of fuel ageing can be potentially harmful to the fuel system.

Tests have shown that fuel deterioration can take place in the fuel supply chain and in the vehicle fuel system. Fuel ageing is accelerated in the presence of heat, oxygen, water, metal ions and other impurities. The products of oxidative ageing have been shown to be corrosive (e.g. organic acids like formic and acetic acids and acids of higher molecular weight). Polymerisation products are also formed and can drop out.

A detailed list of potential problems for FIE systems from FAME is presented in the attachment to this document.

Blends with FAME:

A particular concern is the oxidation stability of FAME blends with sulphur-free diesel fuel (S <10 ppm), which is already available in some parts of Europe and will become more widely used step by step. The oxidation stability of blends as B5 can greatly decrease when using sulphur-free diesel or 15 ppm sulphur diesel such as introduced in U.S.A. in June 2006.

In some countries, introduction of unesterified biogenic fuel is examined as a blending component. FIE manufacturers do not agree to this.

The FIE manufacturers request their customers to support their efforts to obtain good oxidation stability for biodiesel blends worldwide.

**Diesel Fuel Injection Equipment Manufacturers
Common Position Statement on Fatty Acid Methyl Ester (FAME)
Fuels as a Replacement or Extender for Diesel Fuels
January 2007**

The FIE manufacturers position:

FIE manufacturers encourage the development of renewable compression ignition fuels.

Experience to date with Rapeseed Methyl Ester fuels in Europe suggests that RME conforming to the European standard EN14214 at the point of sale used in mixtures of up to 5% by volume with mineral diesel fuel complying with the EN590 diesel fuel standard should not give end-users any serious problems. **The currently agreed position of all FIE manufacturers undersigned is to limit release of injection equipment for admixtures up to a maximum of 5% FAME (meeting the EN14214 standard) with unadulterated diesel fuel (meeting the EN590 standard). The final product B5 must also comply with EN 590.** Any new biodiesel blend, e.g. B 10 in Europe or B 20 in the USA has to be standardised (with special emphasis on oxidation stability) and validated carefully before release.

The required quality of the FAME fuel is defined in European standard EN14214, which covers relevant impurities and tramp chemicals from the processing. Suppliers of FAME fuels must be able to demonstrate compliance to this standard at the filling station. There are several risks associated with possible supply chains.

For the FIE manufacturers a key property of any FAME fuel is the resistance to oxidation. Aged or poor quality FAME contains organic acids like formic and acetic acids and acids of higher molecular weight as well as polymerization products which attack many components, drastically reducing the service life of the FIE. A list of issues which have been witnessed in service is detailed in the attachment to this document.

To date the ASTM specification for FAME fuel (D6751) does not contain a requirement for oxidation stability. A proposal to adopt the EN 14112 Rancimat method with an appropriate limit value for the biodiesel B100 used for blending up to B20 is not yet approved. The FIE manufacturers propose to include an appropriate oxidation stability value in any blend specification (and also ASTM D975 in case it permits blending). The FIE manufacturers are furthermore concerned with the lack of sufficient safeguards against blend quantity.

The FIE manufacturers can accept no legal liability for failure attributable to operating their products with fuels for which the products were not designed, and no warranties or representations are made as to the possible effects of running these products with such fuels.

Non-compliance of the fuel to standards agreed by the FIE manufacturers, whether being evident by appearance of the known degradation products of these fuels, or their known effects within the fuel injection equipment, (see attached list of known issues) will render the FIE Manufacturers' guarantee null & void.

Attachment

Fuel Injection Equipment – Potential Problems with FAME
(non-exhaustive list)

Fuel Characteristic	Effect	Failure Mode
Fatty acid methyl esters (general)	Softening, swelling or hardening and cracking of some elastomers including nitrile rubbers (physical effect depends upon elastomer composition) Displacement of deposits from diesel operation	Fuel leakage Filter plugging
Free methanol in FAME	Corrosion of aluminium & zinc Low flash point	Corrosion of FIE
FAME process chemicals	Entry of potassium & sodium and water hardness (alkaline earth metals) Entry of free fatty acids hastens the corrosion of non ferrous metals, e.g. zinc Salt formation with organic acids (soaps) Sedimentation	Filter plugging Corrosion of FIE Filter plugging Sticking moving parts
Free water	Reversion (Hydrolysis) of FAME to fatty acid and methanol Corrosion Sustainment of bacterial growth Increase of electrical conductivity of the fuel	 Corrosion of FIE Filter plugging
Free glycerine	Corrosion of non-ferrous metals Soaking of cellulose filters Sediment on moving parts and lacquering	Filter plugging Injector coking
Mono-, di- and tri-glyceride	Similar to glycerine	Injector coking
Higher modulus of elasticity	Increase of injection pressure	Potential for reduced service life
High viscosity at low temperature	Generation of excessive heat locally in rotary type distributor pumps Higher stressing of components	Fuel delivery problems Pump seizures Early life failures Poor nozzle spray atomization
Solid impurities / particles	Potential lubricity problems	Reduced service life Nozzle seat wear Blocked nozzles
Ageing products		
Corrosive acids (formic & acetic)	Corrosion of all metal parts May form simple cell	Corrosion of FIE
Higher molecular organic acids	Similar to fatty acid	
Polymerisation products	Deposits, precipitation especially from fuel mixes	Filter plugging Lacquer formation by soluble polymers in hot areas

DELPHI



BOSCH

SIEMENS VDO
A u t o m o t i v e

DENSO

STANADYNE

The views contained in this Common Position Statement are those of the Joint FIE Manufacturers, which comprises the following companies:

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