Standardisation of Vegetable Oils

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ABSTRACT

The paper gives a review of standards dealing with vegetable oils, with particular regard to the use of vegetable oils as raw material for chemical and industrial applications.
The review evidences the availability of a conspicuous number of standardised methods for sampling and analysis of oilseeds, oilseed residues and vegetable oils; these methods are applicable either to food or non-food oils. On the other hand the review remarks the lack of standardised specifications for vegetable oils as raw material, while edible oil specifications are widely present in national standardisation bodies' catalogues and in FAO's Codex Alimentarius. The paper is completed by the presentation of a standard developed for the use of rapeseed oil as fuel, in order to give an example of a non-food vegetable oil standard.

Key-words: vegetable oil, standard, fuel, specification

FOREWORDS

Although natural fats and oils are among the main biological raw material for chemical and technical application (the estimated annual consumption in Europe is about 2,6 million tons), only a limited fraction (according to different estimations between 15 and 25%) of the total vegetable oil supply in the world are used in the industrial sectors, in non-food or technical applications.
The above figures clearly indicate that the food sector still remains the reference point for oil quality and therefore for standards, especially those dealing with specifications and classes.
On the other side it should be taken into account that chemical and technical applications are already a consolidated reality and have a significant potential to increase their importance.
Here are some examples.

Biodegradable Lubricants and Hydraulic Fluids
Only a small fraction (35.000 tonnes) of the European market for lubricants (4,5 million tonnes) is actually derived from vegetable oils, but manufacturers of biodegradable lubricants estimate a potential market for their products much more higher (370.000 tonnes).

Surfactants and Cosmetics
The increasing demand for the use of "natural" ingredients in personal-care products could represent a new potential market for vegetable oil applications.

Fuels
Biodiesel, that uses vegetable oils as raw material, is regarded as a mature technology but still having a market penetration strongly influenced by conventional fossil fuels price. The use of straight vegetable oils as fuel is a fairly recent "rediscovery" that still need to be technically developed and improved. An important factor that will foster the use of vegetable oil and their derivatives as fuels is the European legislation now in preparation concerning the regulatory and fiscal promotion of biofuels.

Linoleum
Main expectances for an increasing linoleum market are related to particular benefits in high-tech situations in being anti-static.

Solvents
Health, security and environmental considerations are expected to be the main drivers for an increase market of vegetable oils based solvents.

Printing Inks
Strong efforts have been made to formulate environmentally friendly printing inks, that, in some cases, have physical properties and performances that meet or exceed the industry standards. These inks appear to be cost competitive with petroleum- based inks and have better environmental properties such as biodegradability, lower VOC, and good deinkability. Some EU members are gradually moving to vegetable oil based inks, but mineral oil-based inks still continue to be used for the most part

Polymers
Although the majority of polymers are derived from petroleum, many polymers experts think there is a good opportunity for vegetable oils to increase their presence in polymer production, either as functional additives, or reactive ingredients or, last but not least, for direct production of polymers.

STANDARDS AND CUSTOMER REQUIREMENTS

Vegetable oils purchased by industries (acting as customers) for non-food purposes, as any other raw material, should meet some requirements, i.e. fulfill needs or expectations that are stated or generally implied. Specified requirements must always be somehow stated, usually in documents called specifications.

Generally speaking, a specification, i.e. a document stating requirements, can be the result of:
- a private agreement between the supplier and the customer;
- a wider agreement that involves different parties (suppliers, customers, other parties) interested in setting up a common specification for repeated and continuous application; such an agreement becomes a standard if approved by a recognised standardisation body either national or international.

What above introduced is important to understand the key factors that lead to standards development: a sufficiently widespread interest and a repeated and continuous application.
If this seems to be the case of the edible oil sector that absorbs more than three quarters of the total vegetable oil supply, it should be clearly assessed if this could also be the case of vegetable oils for industrial and technical applications.

ISO AND CEN STANDARDS DEALING WITH VEGETABLE OILS

The main standardisation bodies involved in the elaboration of standards concerning oilseeds, oilseed residues and vegetable oils are ISO and CEN. ISO and CEN standards deal mainly with methods of test and analysis, while product specifications are usually included in national standards or in the standard provided by FAO's Codex Alimentarius.
It must be underlined that oil specifications are usually conceived for food and feed stuff, while official standards catalogues don't include oil specifications expressly determined for industrial and technical applications.
ISO Standards
ISO/TC 34 "Food products" scope is the standardization in the field of human and animal foodstuffs as well as animal and vegetable propagation materials, in particular terminology, sampling, methods of test and analysis, product specifications and requirements for packaging, storage and transportation. This scope excludes essential oils and starch, which fall respectively under the scope of ISO/TC 54 and ISO/TC 93. ISO/TC 34 has activated the subcommittee "Oleaginous seeds and fruits" (ISO/TC 34/SC2) , which has already elaborated 26 ISO standards, listed in Table 1. ISO/TC 34/SC2 is currently working at
7 new standard listed in Table 2.
It is more specific the scope of ISO/TC 34/SC 11 " Animal and vegetable fats and oils", a Technical Committee which has already prepared 53 ISO standards, listed in Table 3, and which is currently elaborating the work items listed in Table 4

CEN Standards
CEN/TC 307 "Oilseeds, vegetable and animal fats and oils and their by-products - Methods of sampling and analysis" scope is the standardization of methods of sampling and analysis in the fields of oilseeds, vegetable and animal fats and oils and their by-products. This includes physical, physical-chemical and biochemical methods. The main aim of CEN/TC 307 is to

Table 1. ISO standards published under the direct responsibility of ISO/TC 34/SC2 "Oleaginous seeds and fruits"

Number	Title
ISO 542:1990	Oilseeds — Sampling
ISO 658:2002	Oilseeds — Determination of content of impurities
ISO 659:1998	Oilseeds — Determination of oil content (Reference method)
ISO 664:1990	Oilseeds — Reduction of laboratory sample to test sample
ISO 665:2000	Oilseeds — Determination of moisture and volatile matter content
ISO 729:1988	Oilseeds — Determination of acidity of oils
ISO 734-1:1998	Oilseed residues — Determination of oil content — Part 1: Extraction method with hexane (or light petroleum)
ISO 734-2:1998	Oilseed residues — Determination of oil content — Part 2: Rapid extraction method
ISO 735:1977	Oilseed residues — Determination of ash insoluble in hydrochloric acid
ISO 749:1977	Oilseed residues — Determination of total ash
ISO 771:1977	Oilseed residues — Determination of moisture and volatile matter content
ISO 5500:1986	Oilseed residues — Sampling
ISO 5502:1992	Oilseed residues — Preparation of test samples
ISO 5506:1988	Soya bean products — Determination of urease activity
ISO 5507:2002	Oilseeds, vegetable oils and fats — Nomenclature
ISO 5511:1992 (+Corr.1:1997)	Oilseeds — Determination of oil content — Method using continuous-wave low-resolution nuclear magnetic resonance spectrometry (Rapid method)
ISO 7700-2:1987	Check of the calibration of moisture meters — Part 2: Moisture meters for oilseeds
ISO 8892:1987	Oilseed residues — Determination of total residual hexane
ISO 9167-1:1992	Rapeseed — Determination of glucosinolates content — Part 1: Method using high-performance liquid chromatography
ISO 9167-2:1994	Rapeseed — Determination of glucosinolates content — Part 2: Method using X-ray fluorescence spectrometry
ISO 9289:1991	Oilseed residues — Determination of free residual hexane
ISO 10519:1997	Rapeseed — Determination of chlorophyll content — Spectrometric method
ISO 10565:1998	Oilseeds — Simultaneous determination of oil and water contents — Method using pulsed nuclear magnetic resonance spectrometry
ISO 10632:2000	Oilseed residues — Simultaneous determination of oil and water contents — Method using pulsed nuclear magnetic resonance spectroscopy
ISO 10633-1:1995	Oilseed residues — Determination of glucosinolates content — Part 1: Method using high-performance liquid chromatography

Table 2. ISO standards in preparation under the direct responsibility of ISO/TC 34/SC2 "Oleaginous seeds and fruits"

Number	Title
ISO/DIS 734-1	Oilseed residues — Determination of oil content — Part 1: Extraction method with hexane (or light petroleum)
ISO/DIS 734-2	Oilseed residues — Determination of oil content — Part 2: Rapid extraction method
ISO/AWI 7700-2	Checking the performance of moisture meters in use — Part 2: Moisture meters for oilseeds
ISO/CD 9167-1	Rapeseed — Determination of glucosinolate content — Part 1: Method using high-performance liquid chromatography
ISO/AWI 9167-3	Rapeseed — Determination of glucosinolate content — Part 3: Method using isocratic high-performance liquid chromatography
ISO/AWI 9167-4	Rapeseed — Determination of glucosinolate content — Part 4: Spectrometric method for total glucosinolates by glucose release
ISO/AWI 16800	Oilseeds and oilseed residues — Simplified method for determination of the moisture and volatile matter content

harmonise European methods with international methods; priority is given to the adoption of ISO methods.

Therefore CEN standards already published are only adoptions of ISO standards, while the actual Work Programme (Table 5) includes also the development of original FAME standards, which are part of a wider standardisation program on biodiesel which involves also CEN/TC 19.

Table 3. ISO standards published under the direct responsibility of ISO/TC 34/SC11 "Animal and vegetable fats and oils"

Number	Title the main element of the title is always "Animal and vegetable fats and oils"
ISO 660:1996	Determination of acid value and acidity
ISO 661:1989	Preparation of test sample
ISO 662:1998	Determination of moisture and volatile matter content
ISO 663:2000	Determination of insoluble impurities content
ISO 934:1980	Determination of water content — Entrainment method
ISO 935:1988	Determination of titre
ISO 3596:2000	Determination of unsaponifiable matter — Method using diethyl ether extraction
ISO 3656:2002	Determination of ultraviolet absorbance expressed as specific UV extinction
ISO 3657:2002	Determination of saponification value
ISO 3960:2001	Determination of peroxide value
ISO 3961:1996	Determination of iodine value
ISO 5508:1990	Analysis by gas chromatography of methyl esters of fatty acids
ISO 5509:2000	Preparation of methyl esters of fatty acids
ISO 5555:2001	Sampling
ISO 5558:1982	Detection and identification of antioxidants — Thin-layer chromatographic method
ISO 6320:2000	Determination of refractive index
ISO 6321:2002	Determination of melting point in open capillary tubes (slip point)
ISO 6463:1982	Determination of butylhydroxyanisole (BHA) and butylhydroxytoluene (BHT) — Gas-liquid chromatographic method
ISO 6464:1983	Determination of gallates content — Molecular absorption spectrometric method
ISO 6656:2002	Determination of polyethylene-type polymers
ISO 6800:1997	Determination of the composition of fatty acids in the 2-position of the triglyceride molecules
ISO 6883:2000	Determination of conventional mass per volume ("litre weight in air")
ISO 6884:1985	Determination of ash
ISO 6885:1998	Determination of anisidine value
ISO 6886:1996	Determination of oxidation stability (Accelerated oxidation test)
ISO 7366:1987	Determination of 1-monoglycerides and free glycerol contents
ISO 7847:1987	Determination of polyunsaturated fatty acids with a cis,cis 1,4-diene structure
ISO 8292:1991	Determination of solid fat content — Pulsed nuclear magnetic resonance method
ISO 8294:1994	Determination of copper, iron and nickel contents — Graphite furnace atomic absorption method
ISO 8420:2002	Determination of content of polar compounds
ISO 8534:1996	Determination of water content — Karl Fischer method
ISO 9832:2002	Determination of residual technical hexane content
ISO 9936:1997	Determination of tocopherols and tocotrienols contents — Method using high-performance liquid chromatography
ISO 10539:2002	Determination of alkalinity
ISO 10540-1:2003	Determination of phosphorus content — Part 1: Colorimetric method
ISO 10540-2:2003	Determination of phosphorus content — Part 2: Method using graphite furnace atomic absorption spectrometry
ISO 10540-3:2002	Determination of phosphorus content — Part 3: Method using inductively coupled plasma (ICP) optical emission spectroscopy
ISO 12193:1994	Determination of lead content — Graphite furnace atomic absorption method
ISO 12228:1999	Determination of individual and total sterols contents — Gas chromatographic method
ISO 15267:1998	Flashpoint limit test using Pensky-Martens closed cup flash tester
ISO 15301:2001	Determination of sediment in crude fats and oils — Centrifuge method
ISO 15302:1998	Determination of benzo[a]pyrene content — Reverse-phase high-performance liquid chromatography method
ISO 15303:2001	Detection and identification of a volatile organic contaminant by GC/MS
ISO 15304:2002	Determination of the content of trans fatty acid isomers of vegetable fats and oils — Gas chromatographic method
ISO 15305:1998	Determination of Lovibond colour
ISO 15774:2000	Determination of cadmium content by direct graphite furnace atomic absorption spectrometry
ISO 15788-1:1999	Determination of stigmastadienes in vegetable oils — Part 1: Method using capillary-column gas chromatography (Reference method)
ISO 15788-2:2003	Determination of stigmastadienes in vegetable oils — Part 2: Method using high-performance liquid chromatography (HPLC)
ISO 16035:2003	Determination of low-boiling halogenated hydrocarbons in edible oils
ISO 16931:2001	Determination of polymerized triglycerides by high-performance size-exclusion chromatography (HPSEC)
ISO 18609:2000	Determination of unsaponifiable matter — Method using hexane extraction
ISO 19219:2002	Determination of visible foots in crude fats and oils

Table 4. ISO standards in preparation under the direct responsibility of ISO/TC 34/SC11 "Animal and vegetable fats and oils"

Number	Title the main element of the title is always "Animal and vegetable fats and oils"
ISO 661	Preparation of test sample
ISO/DIS 12193	Determination of lead by direct graphite furnace atomic absorption spectroscopy
ISO/FDIS 13884	Determination of isolated trans isomers by infrared spectrometry
ISO/CD 15753	Determination of polycyclic aromatic hydrocarbons
ISO/CD 22508	Determination of the relative composition of free fatty acids, mono-, di- and triacylglycerides by gas chromatography — Carbon number analysis

Table 5. CEN/TC 307 Work Programme (the list doesn't include work items dealing with ISO standards adoptions)

Project Reference	Title	Current Status
prEN 14103	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of ester and linolenic acid methyl ester contents	Ratified
prEN 14104	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of acid value	Ratified
prEN 14105	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of free and total glycerol and mono-, di-, triglyceride contents (Reference method)	Ratified
prEN 14107	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of phosphorus content by inductively coupled plasma (ICP) emission spectrometry	Ratified
prEN 14108	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of sodium content by atomic absorption spectrometry	Ratified
prEN 14109	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of potassium content by atomic absorption spectrometry	Ratified
prEN 14110	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of methanol content	Ratified
prEN 14111	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of iodine value	Ratified
prEN 14112	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of oxidation stability (accelerated oxidation test)	Ratified
prEN 14106	Fat and oil derivatives - Fatty Acid Methyl Esters (FAME) - Determination of free glycerol content	Ratified

CEN/TC 19 "Petroleum products, lubricants and related products" has already ratified the standards concerning FAME requirements and test methods : prEN 14213 Heating fuels - Fatty acid methyl esters (FAME) - Requirements and test methods, prEN 14214 Automotive fuels - Fatty acid methyl esters (FAME) for diesel engines - Requirements and test methods.

THE BAVARIAN STANDARD FOR RAPESEED OIL AS FUEL

The Bavarian regional standard for rapeseed oil as a fuel, *Vorläufiger Qualitätsstandard für Rapsöl als Kraftstoff (RK-Qualitätsstandard),* represents an interesting example of a standard specifically conceived for technical applications of vegetable oils, in this case as fuel.

The standard (Table 6) is actually an interesting combination of properties used to characterise vegetable oils in general (iodine value, acid value, etc.) and properties dealing with fuels.

The standard was proposed by the Bavarian National Institute for Agricultural Engineering and the Technical University of Munich Weihenstephan with the aim to allow for provision of a standard oil fuel which would allow for reliable operation of vegetable oil engines in vehicles and to serve as basis for future engine developments.

Table 6. Quality standard for rapeseed oil as a fuel (RK - Qualitätsstandard)

Properties /Contents	Unit	Limiting Value		Testing Method
		min.	max.	
Characteristic properties for Rapeseed Oil				
Density (15ºC)	kg/m³	900	930	DIN EN ISO 3675 DIN EN ISO 12185
Flash Point by P.-M.	ºC	220	-	DIN EN 22719
Calorific Value	kJ/kg	35000	-	DIN 51900-3
Kinematic Viscosity (40ºC)	mm²/S	-	38	DIN EN ISO 3104
Low Temperature Behaviour	-	-	-	Rotational Viscometer (testing conditions will be developed)
Cetane Number	-	-	-	Testing method will be reviewed
Carbon Residue	Mass-%	-	0.40	DIN EN ISO 10370
Iodine Number	g/100 g	100	120	DIN 53241-1
Sulphur Content	mg/kg	-	20	ASTM D5453-93
Variable properties				
Contamination	mg/kg	-	25	DIN EN 12662
Acid Value	mg KOH/g	-	2.0	DIN EN ISO 660
Oxidation Stability (110ºC)	h	5.0	-	IS0 6886
Phosphorus Content	mg/kg	-	15	ASTM D3231-99
Ash Content	Mass-%	-	0.01	DIN EN ISO 6245
Water Content	Mass-%	-	0.075	prEN ISO 12937

CONCLUSIONS

The review of official available standards introduced in the above paragraphs denotes:

a) the availability of a conspicuous number of standardised methods for sampling and analysis of oilseeds, oilseed residues and vegetable oils; these methods are applicable either to food or non-food oils;
b) the lack of standardised specifications for vegetable oils as raw material, while edible oil specifications are widely present in national standardisation bodies' catalogues and in FAO's Codex Alimentarius.

This can witness:

– the difficulty to establish common requirements for different industrial applications;
– the insufficient interest and/or strength of a single industrial sector to establish and consolidate his raw material requirements.
– the difficulty in making vegetable oil suppliers and the whole vegetable oil chain accept standardised specifications for particular applications, considering that the oil chain is now mainly oriented toward the food sector.

Therefore if we want to think about standardisation for non-food vegetable oils, we should first consider if, from a technical and economical point of view, standards are needed, possible and acceptable.

A typical approach of CEN in order to evaluate the opportunity of a new standardisation activity is the creation of a working group or a task force constituted by representatives from interested parties of the sector that cooperates to make a preliminary assessment of what above.

Moreover an important standardisation impulse could come from policymakers that recognize the importance of standards to reach particular objectives.

This has been the case of other sectors where the European Commission has given a standardisation mandate, i.e. a political request addressed to CEN in support of an action from the EC. This can be in support of legislative work such as a directive, or in support of an industrial policy action from the EC.

NORME DI STANDARDIZZAZIONE DEGLI OLI VEGETALI

Il contributo presenta una rassegna delle norme tecniche relative agli oli vegetali, con particolare riguardo all'utilizzo di questi ultimi come materia prima in applicazioni tecniche o industriali.
Si evidenzia la disponibilità di un cospicuo numero di metodi normalizzati per il campionamento di semi oleosi, residui degli stessi e oli vegetali; questi metodi sono applicabili sia nel settore alimentare sia in altri settori. La rassegna normativa rileva altresì la presenza di numerose specifiche normalizzate per gli oli vegetali destinati all'utilizzo alimentare, sia nei repertori degli enti di normazione nazionali sia e soprattutto nel Codex Alimentarius della FAO; al contrario sottolinea la mancanza di riferimenti normativi per gli oli destinati destinati ad utilizzi diversi da quello alimentare. La presentazione di una norma tecnica relativa alle specifiche dell'olio di colza per il suo utilizzo come combustibile completa questo contributo al fine di fornire un esempio di una norma tecnica sull'olio vegetale come combustibile.